

Supplementary Information

New Highly Oxygenated Germacrano^{lides} from *Carpesium divaricatum* and their Cytotoxic Activity

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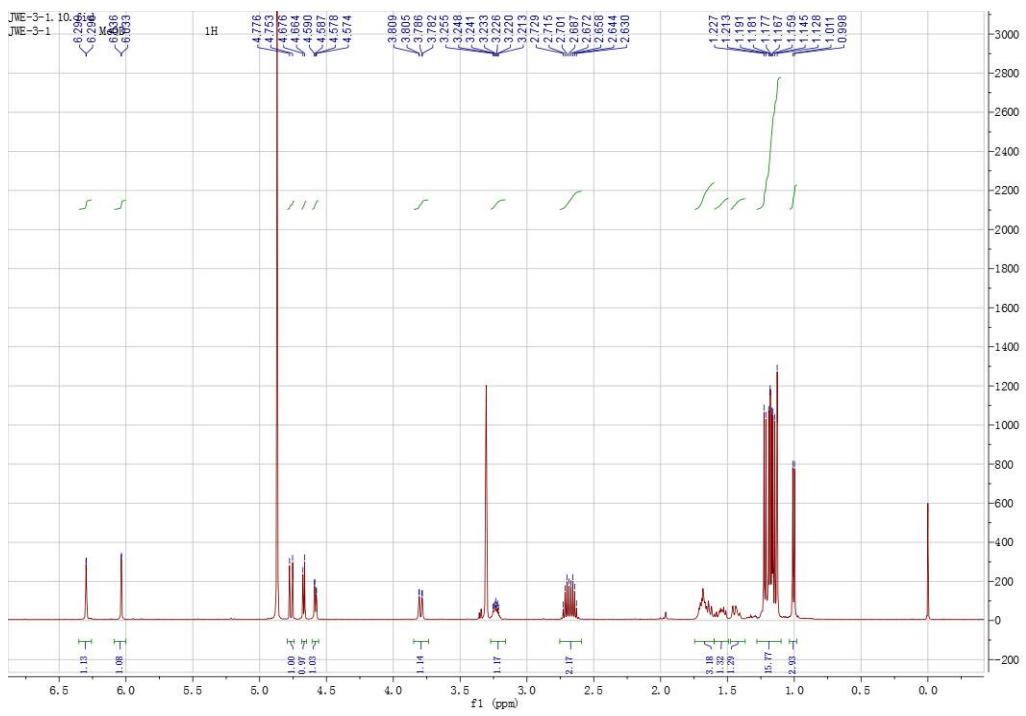


Fig. S1.1 ^1H NMR spectrum (500 MHz) of incaspitolide A (**1**) in CD_3OD

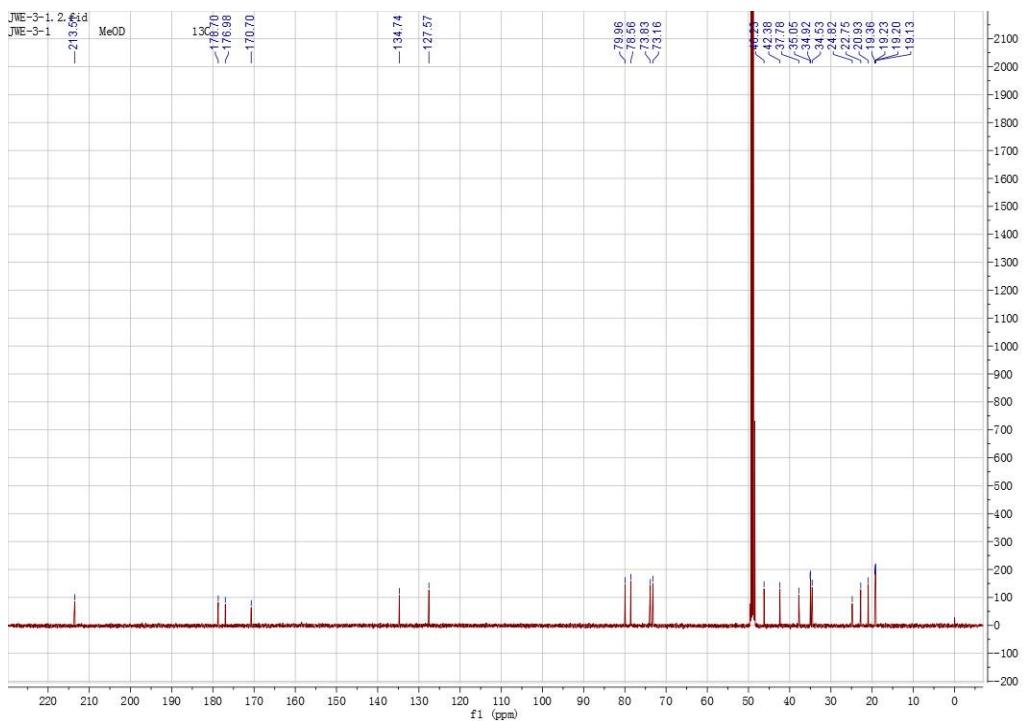


Fig. S1.2 ^{13}C NMR spectrum (500 MHz) of incasitolide A (**1**) in CD_3OD

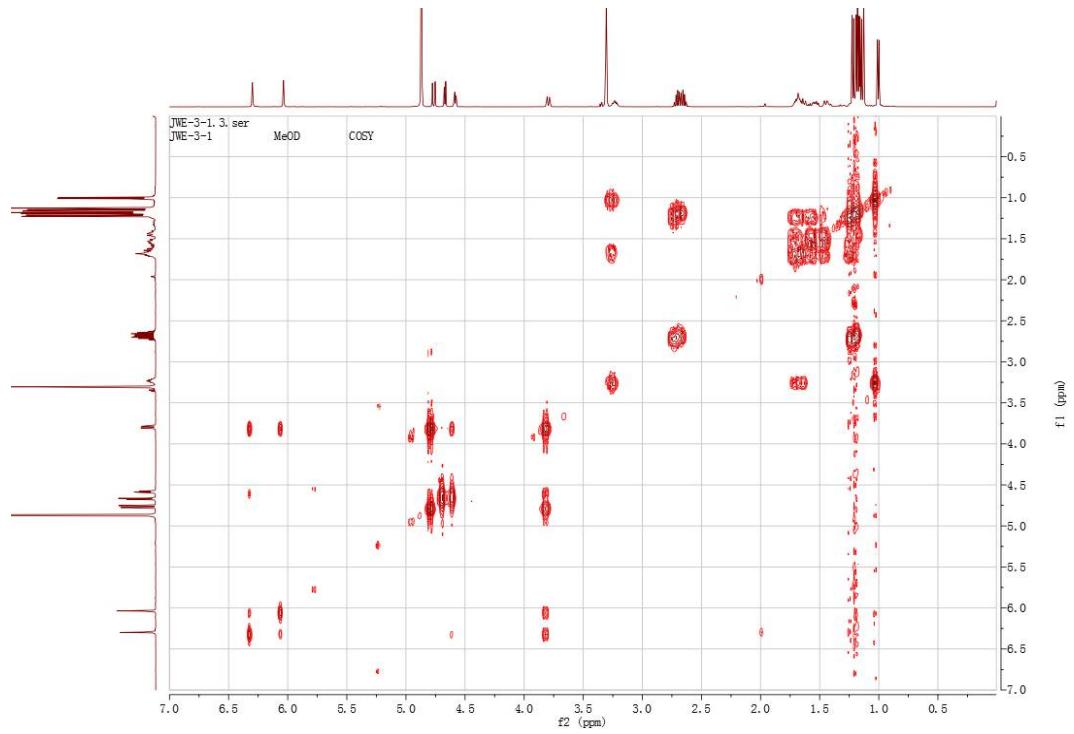


Fig. S1.3 ^1H - ^1H COSY spectrum (500 MHz) of incaspitolide A (**1**) in CD_3OD

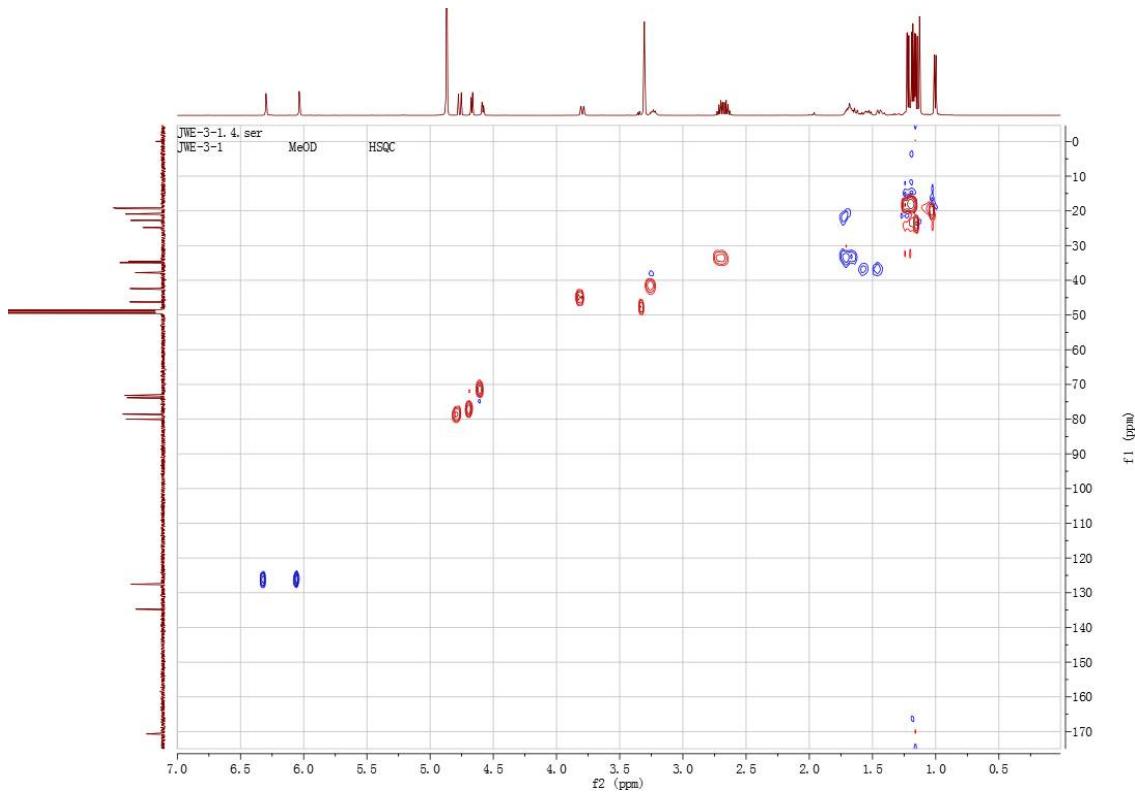


Fig. S1.4 HSQC spectrum (500 MHz) of incaspitolide A (**1**) in CD_3OD

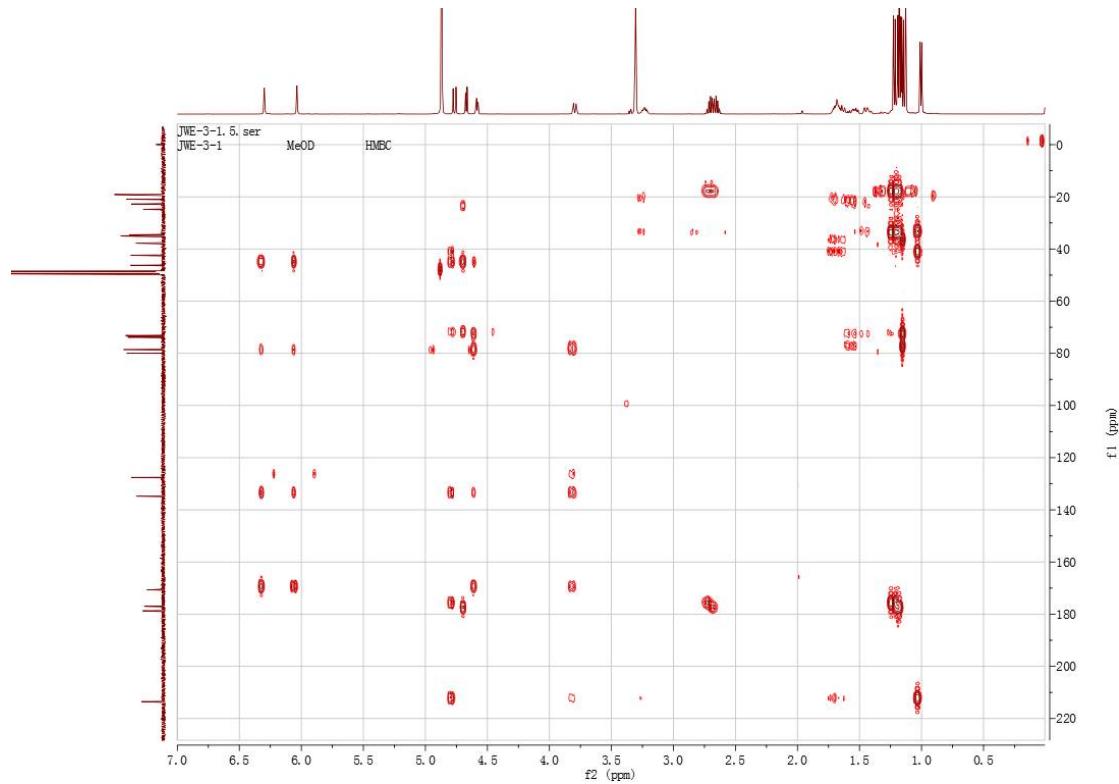


Fig. S1.5 HMBC spectrum (500 MHz) of incaspitolide A (**1**) in CD_3OD

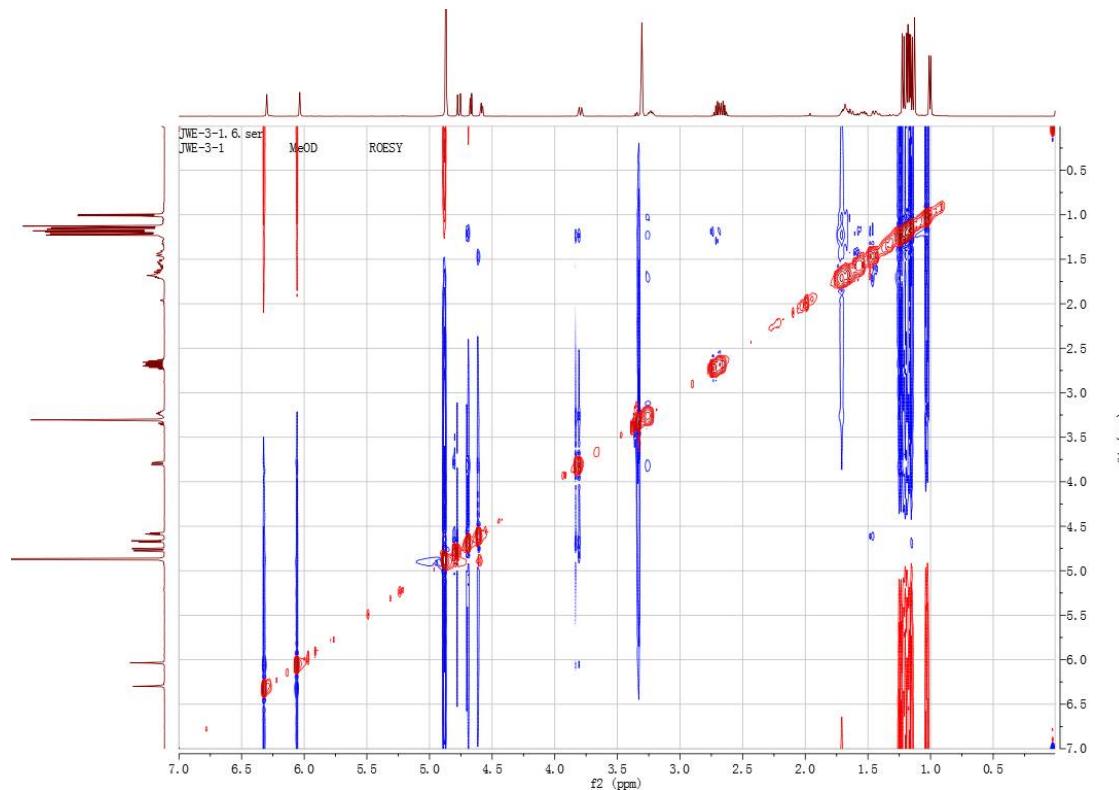
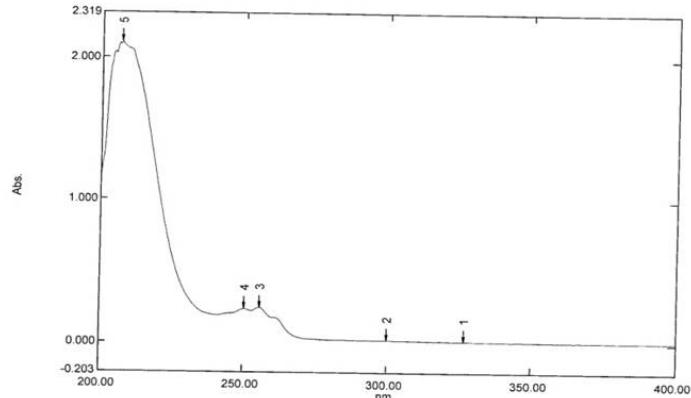


Fig. S1.6 ROESY spectrum (500 MHz) of incaspitolide A (**1**) in CD_3OD

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Fig. S1.7 UV spectrum of incaspitolide A (1)

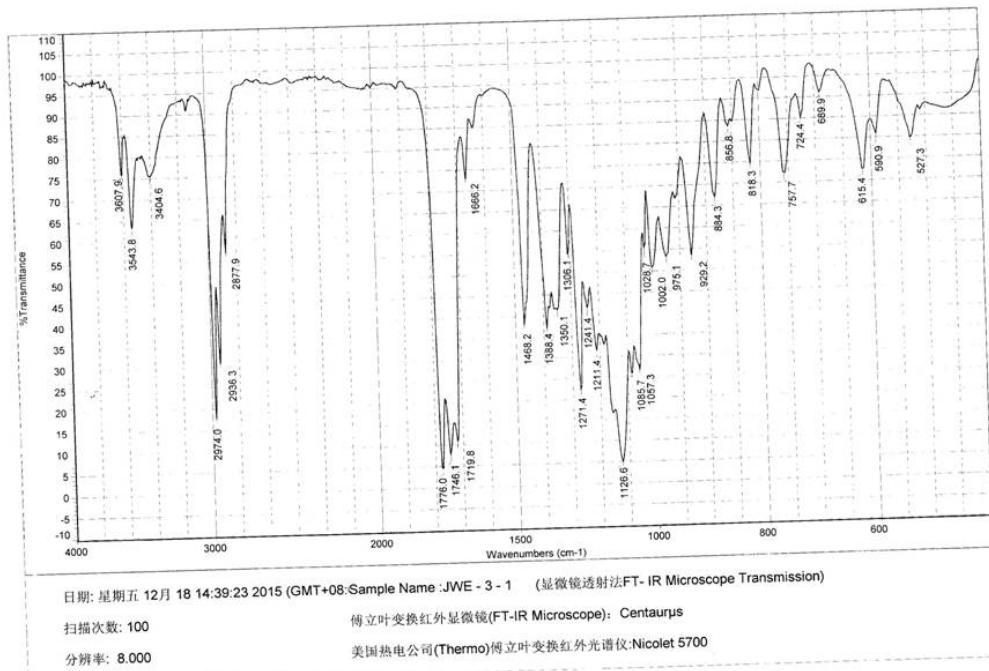


Fig. S1.8 IR spectrum of incaspitolide A (1)

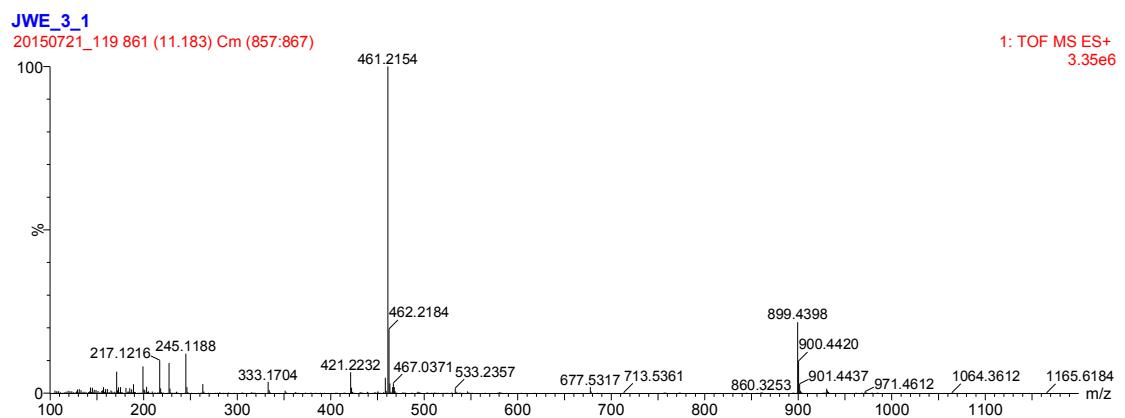


Fig. S1.9 HRESIMS spectrum of incasitolide A (**1**)

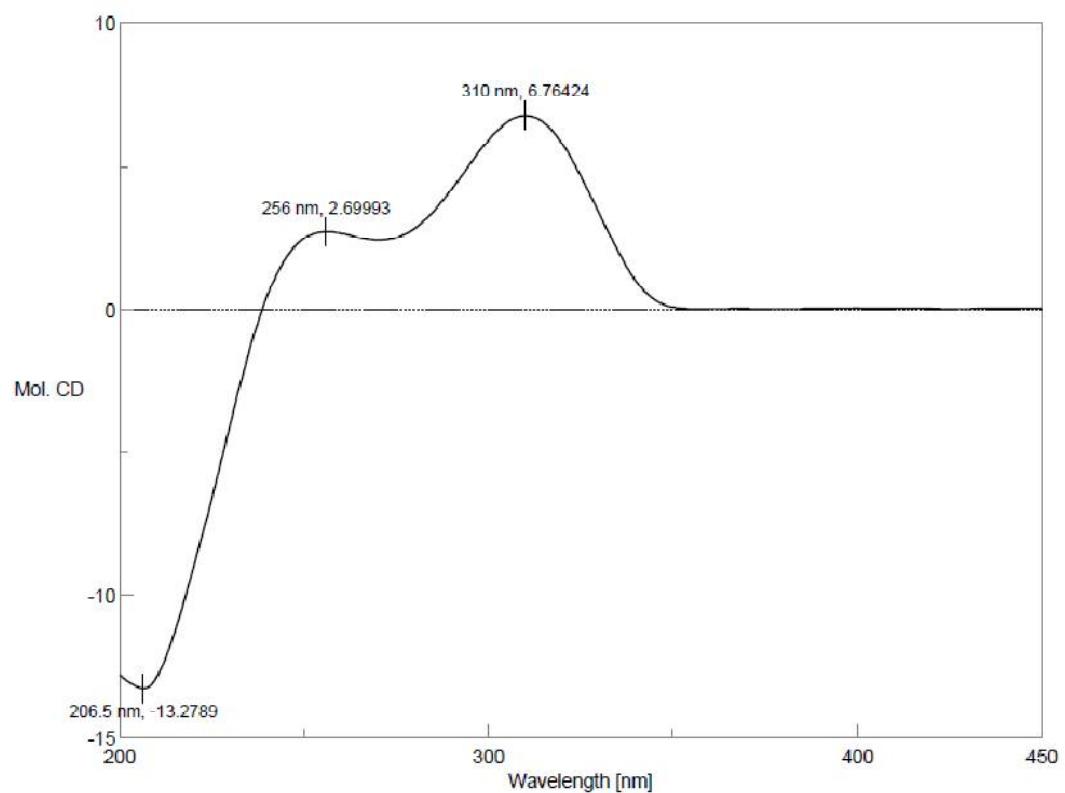


Fig. S1.10 CD spectrum of incasitolide A (**1**)

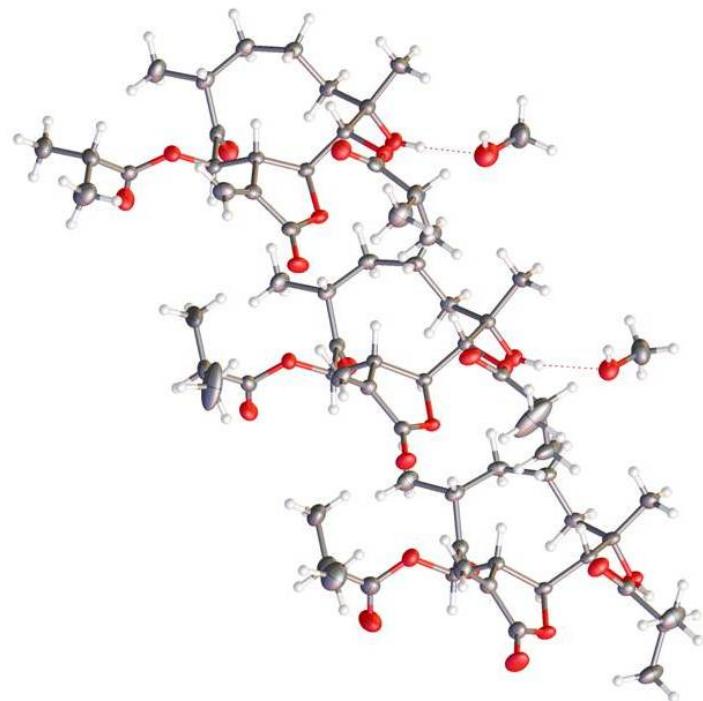


Fig. S1.11 X-ray ORTEP drawing of incaspitolide A (**1**)

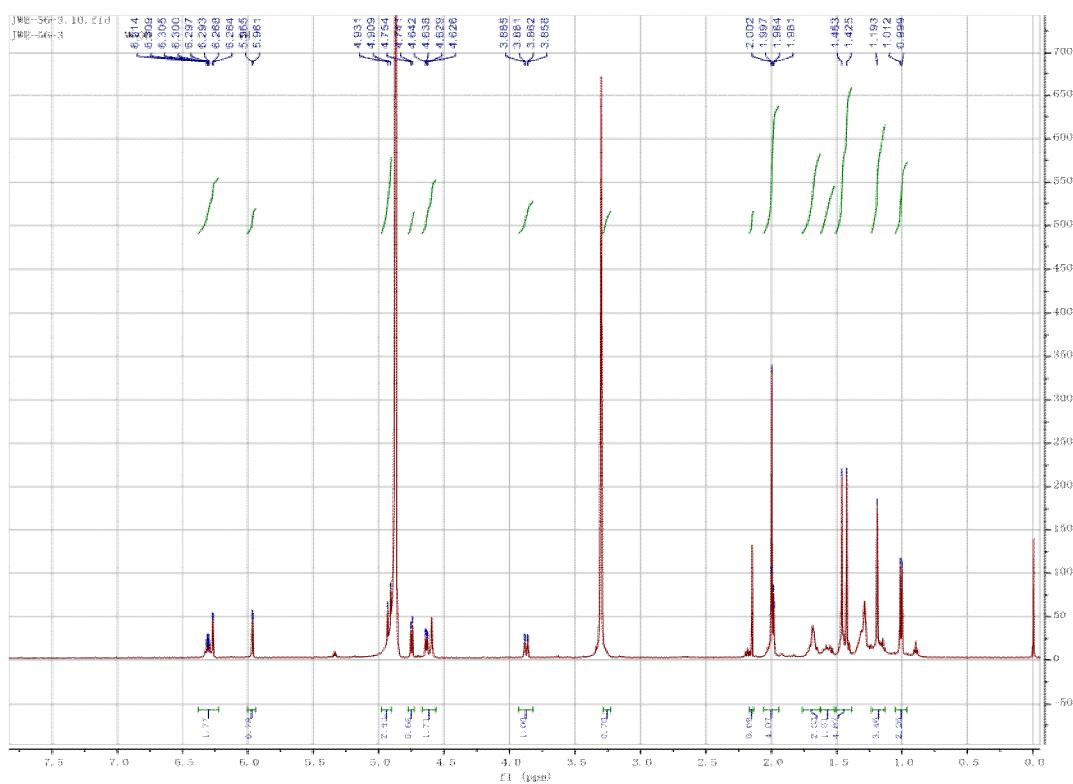


Fig. S2.1 ^1H NMR spectrum (500 MHz) of divarolide A (**2**) in CD_3OD

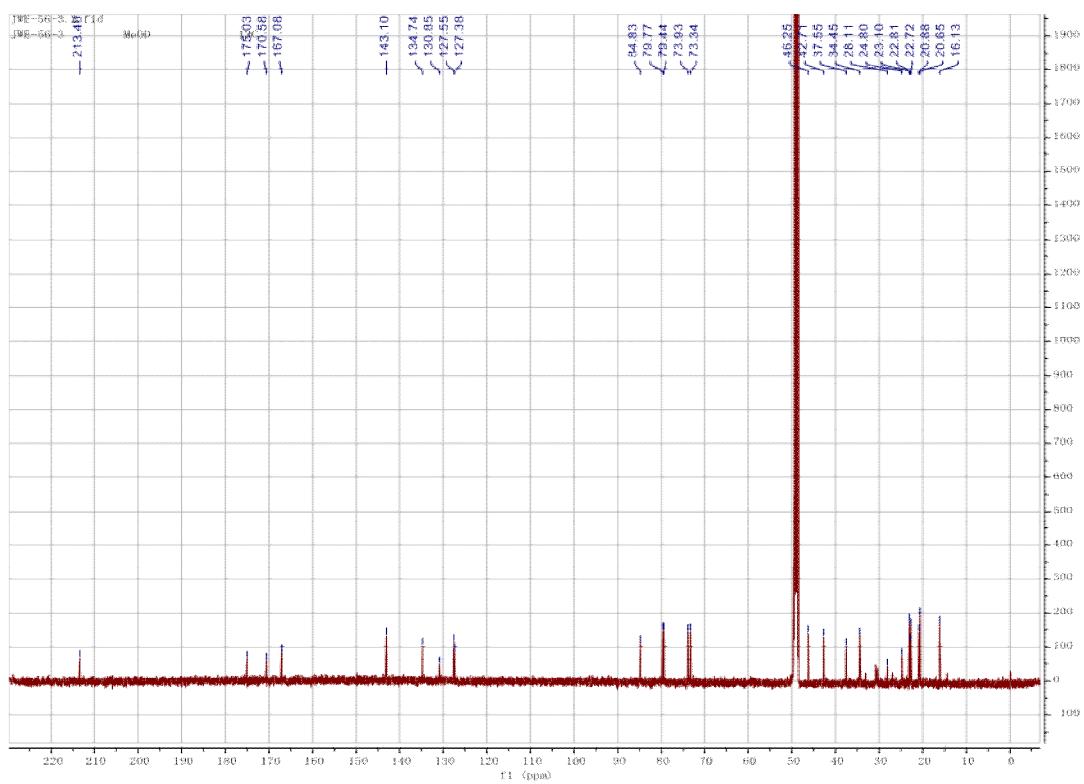


Fig. S2.2 ^{13}C NMR spectrum (500 MHz) of divarolide A (**2**) in CD_3OD

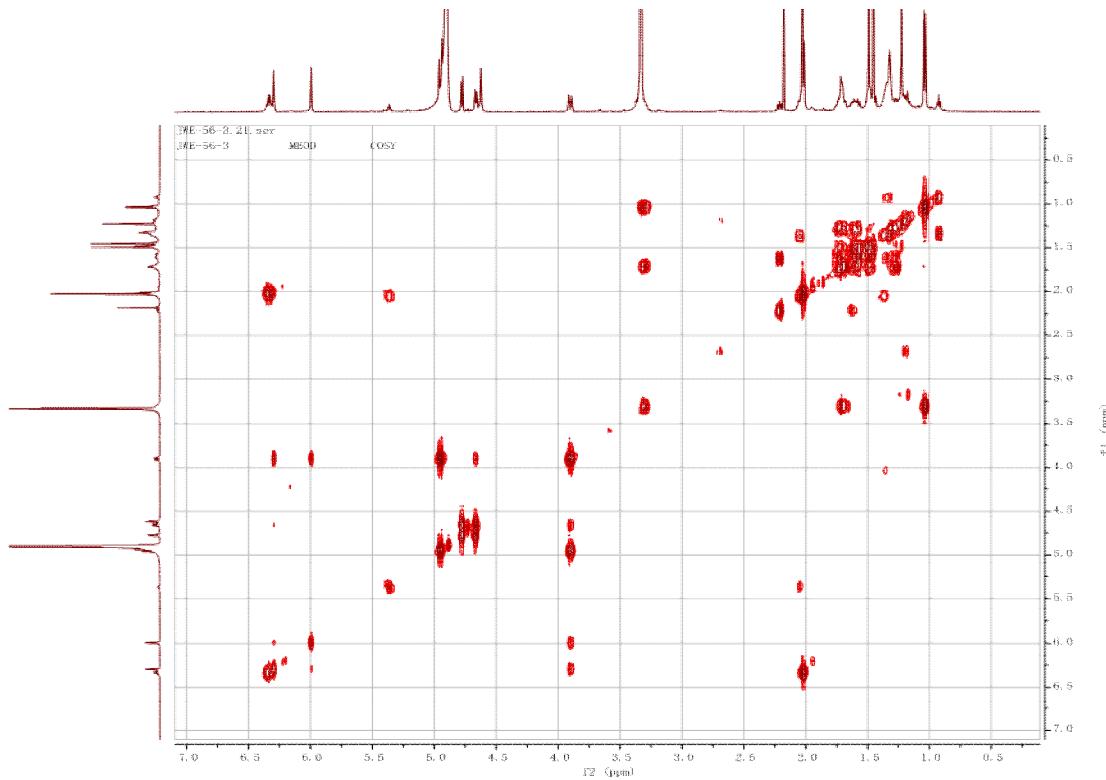


Fig. S2.3 ^1H - ^1H COSY spectrum (500 MHz) of divarolide A (**2**) in CD_3OD

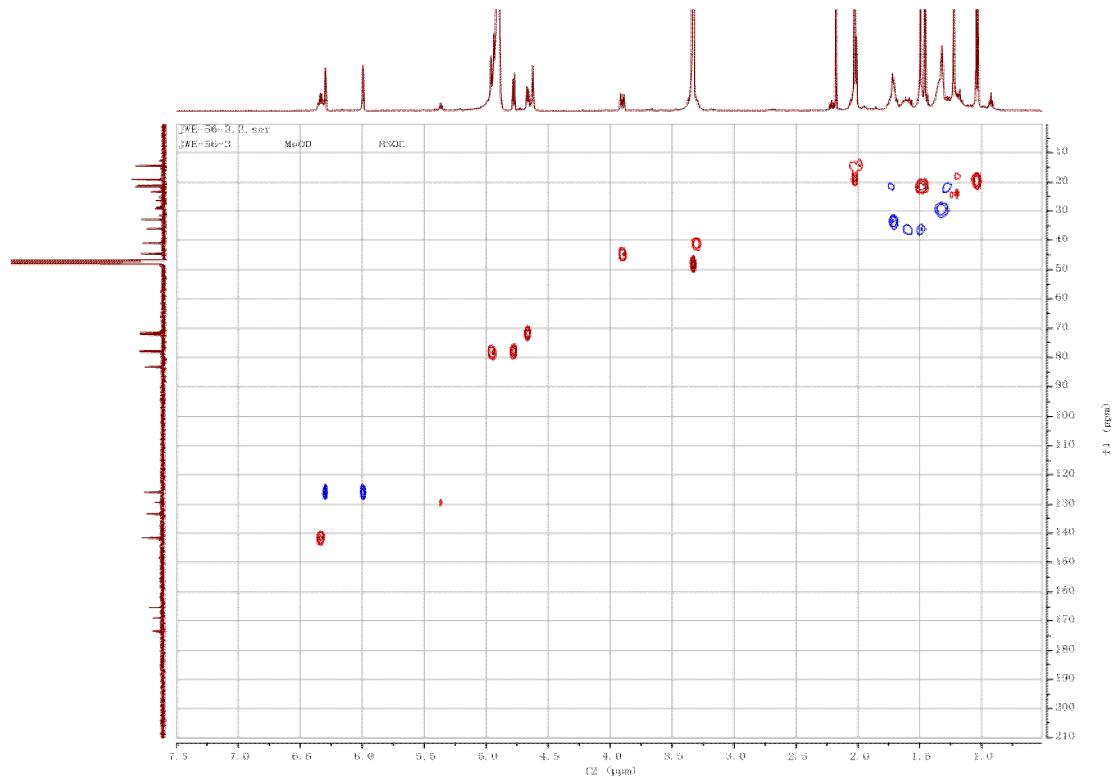


Fig. S2.4 HSQC spectrum (500 MHz) of divarolide A (**2**) in CD_3OD

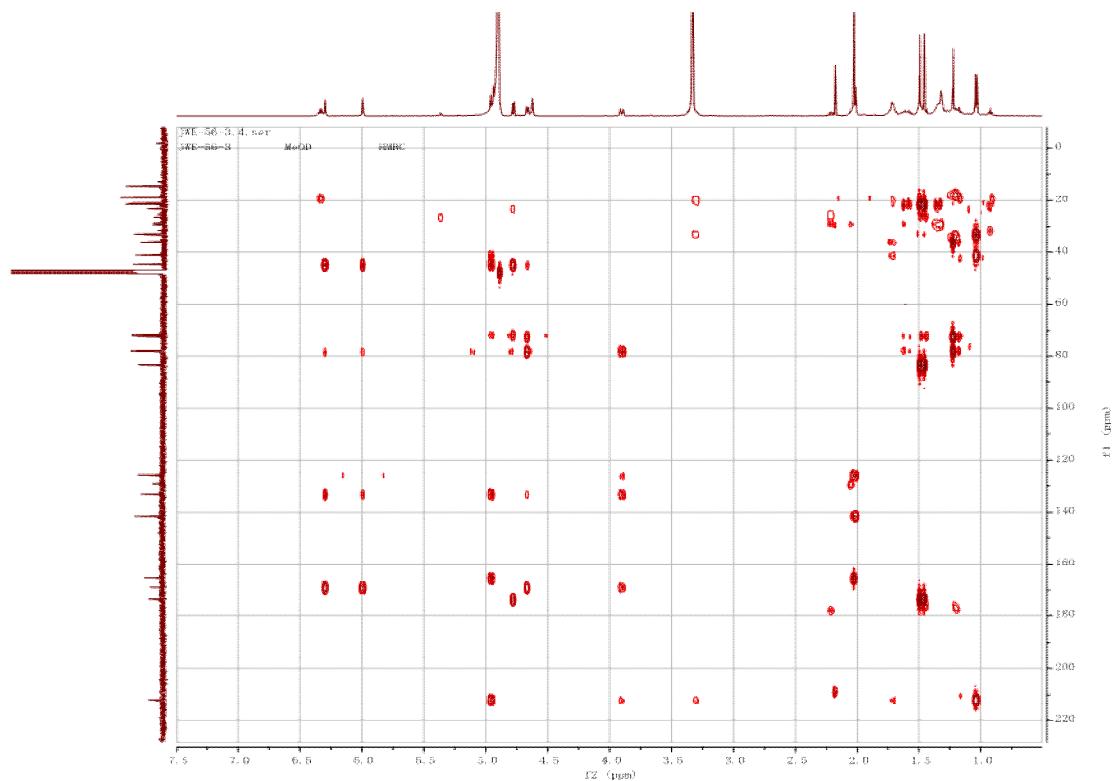


Fig. S2.5 HMBC spectrum (500 MHz) of divarolide A (**2**) in CD_3OD

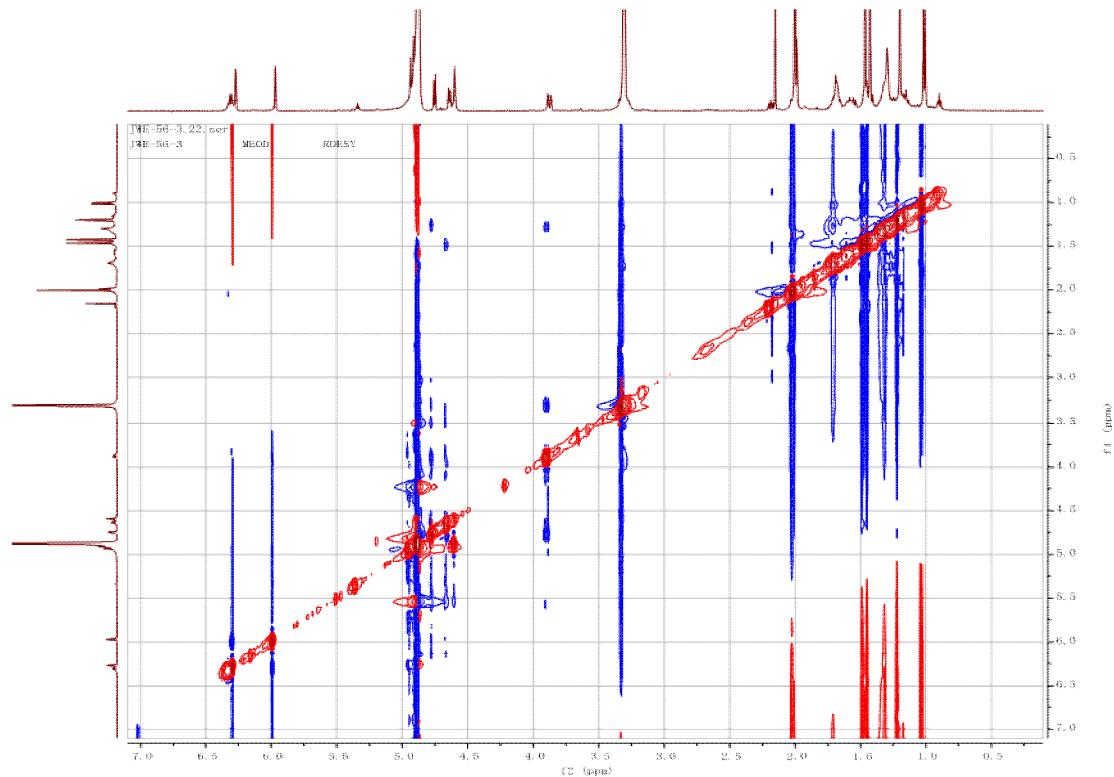
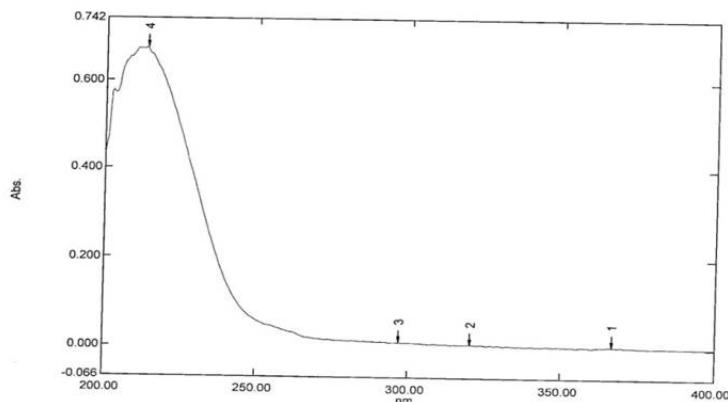


Fig. S2.6 ROESY spectrum (500 MHz) of divarolide A (**2**) in CD_3OD

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2015-12-16 16:02:14

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Fig. S2.7 UV spectrum of divarolide A (**2**)

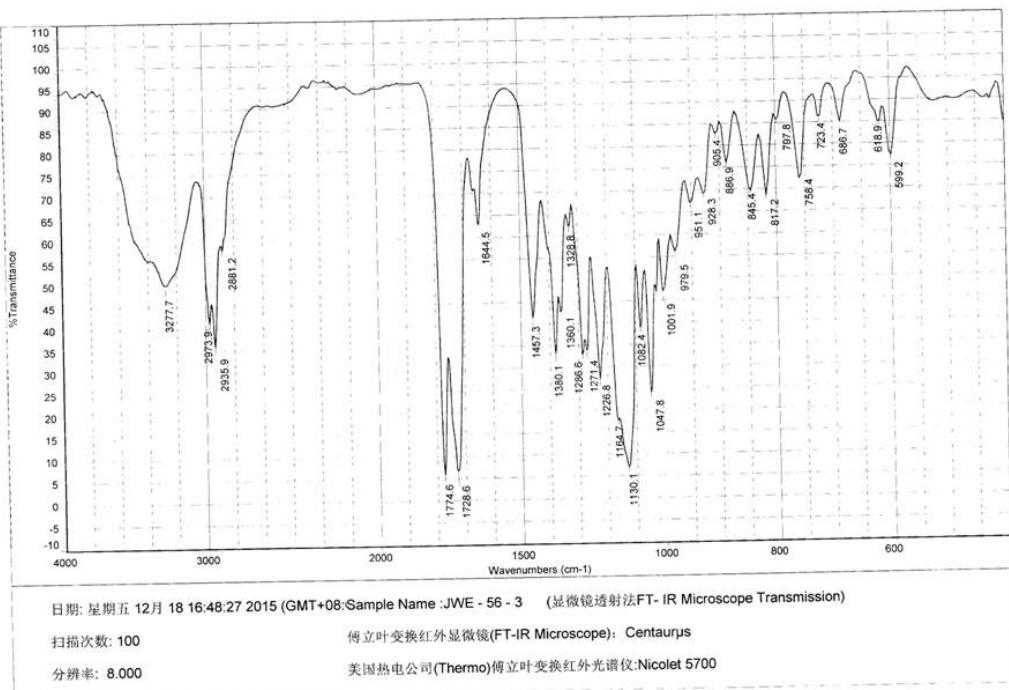


Fig. S2.8 IR spectrum of divarolide A (2)

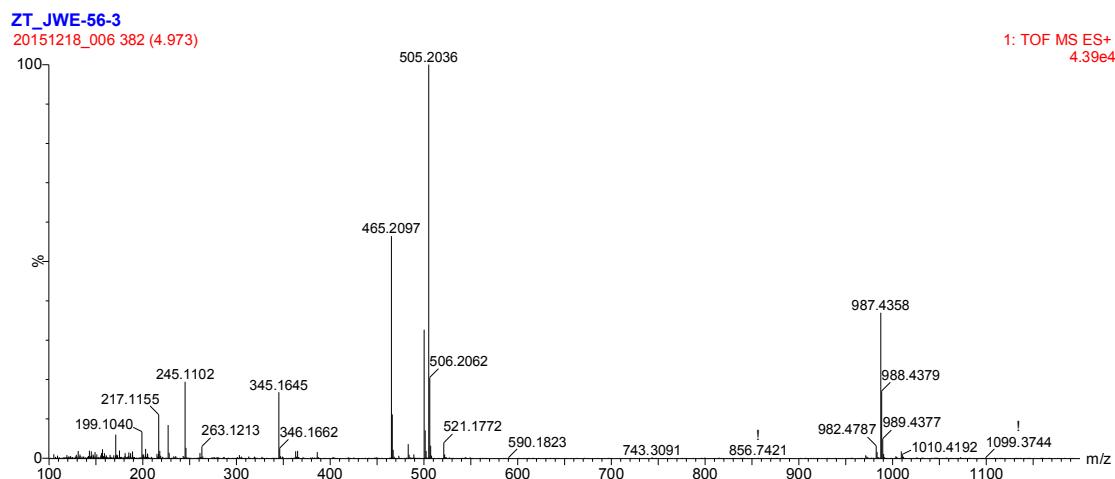


Fig. S2.9 HRESIMS spectrum of divarolide A (2)

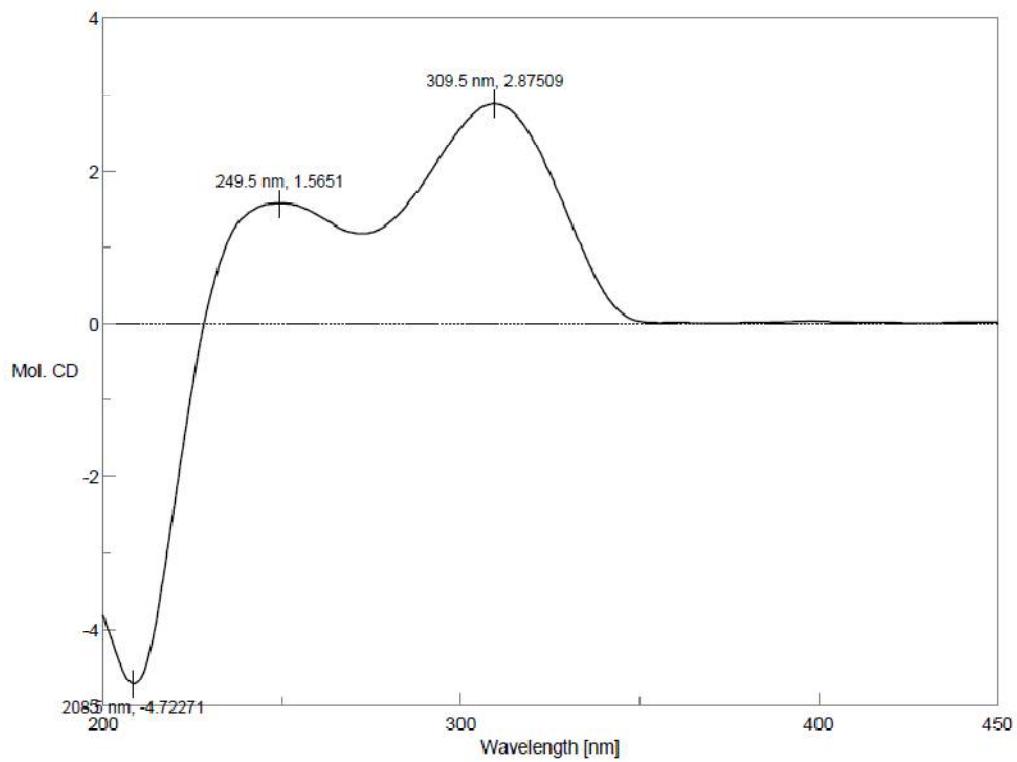


Fig. S2.10 CD spectrum of divarolide A (**2**)

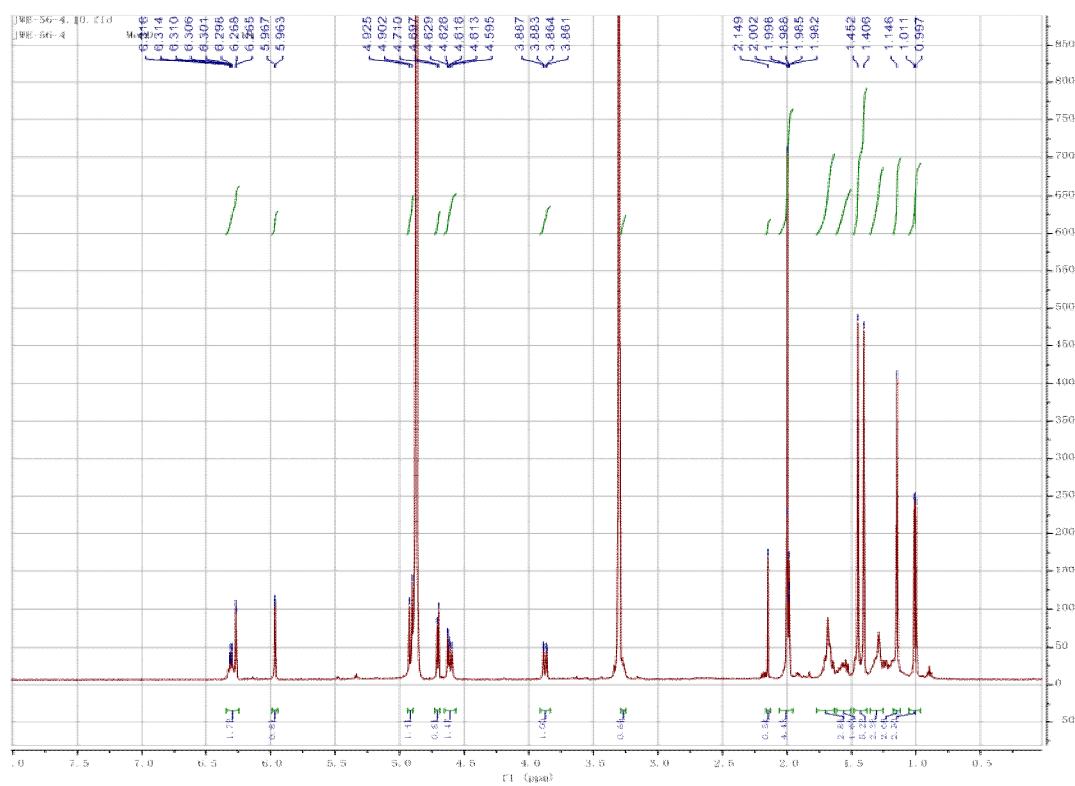


Fig. S3.1 ^1H NMR spectrum (500 MHz) of divarolide B (**3**) in CD_3OD

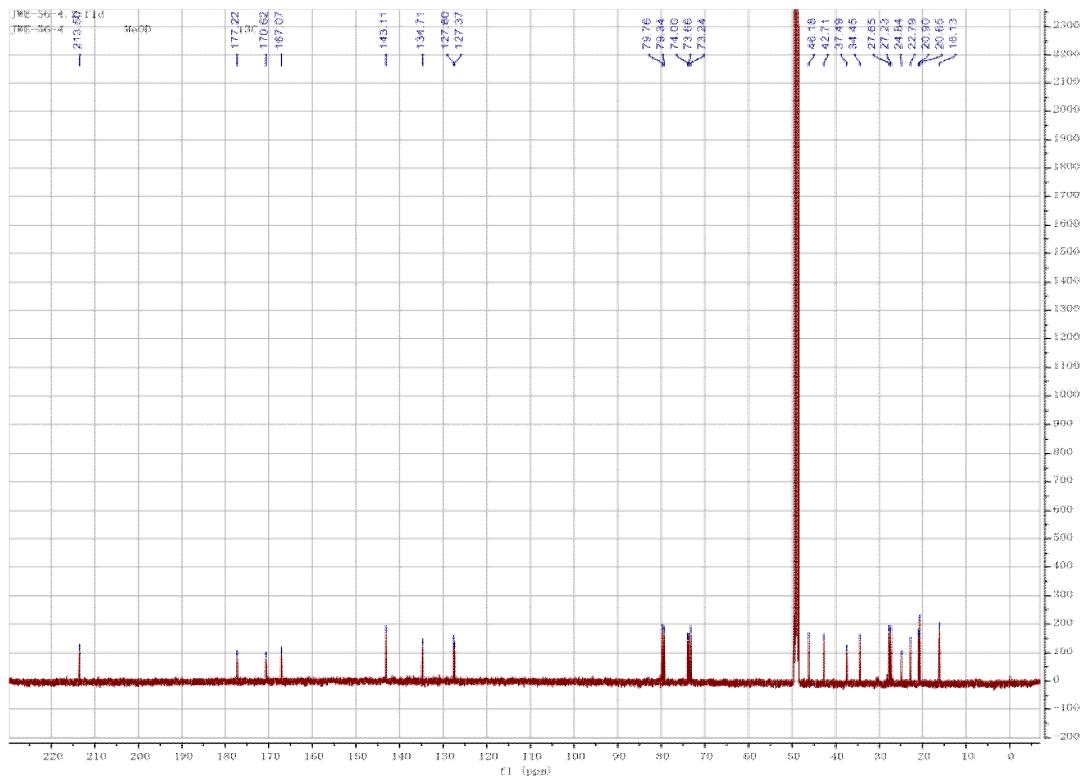


Fig. S3.2 ^{13}C NMR spectrum (500 MHz) of divarolide B (**3**) in CD_3OD

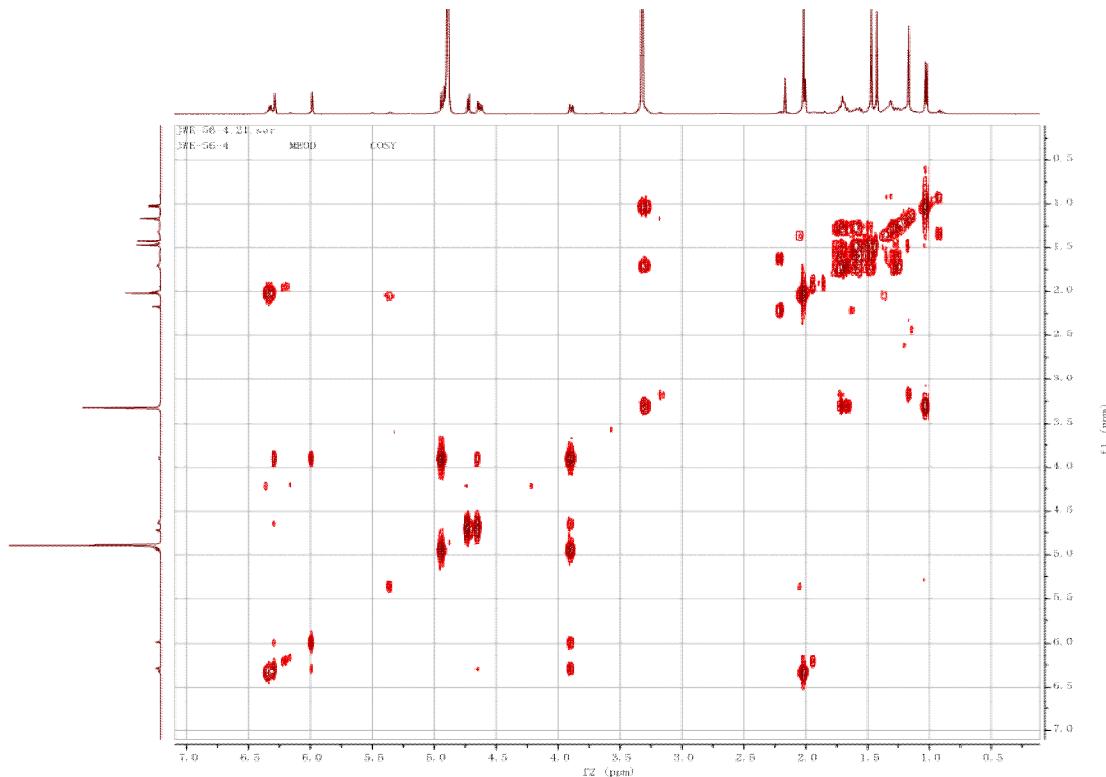


Fig. S3.3 ^1H - ^1H COSY spectrum (500 MHz) of divarolide B (**3**) in CD_3OD

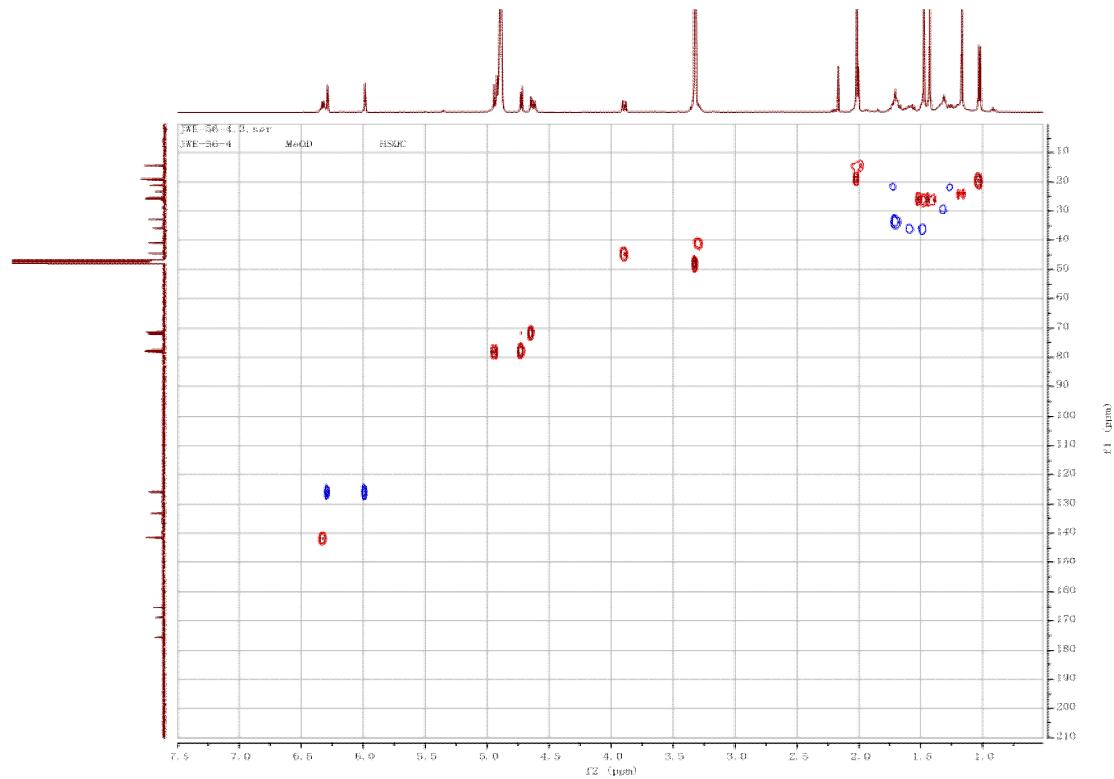


Fig. S3.4 HSQC spectrum (500 MHz) of divarolide B (**3**) in CD_3OD

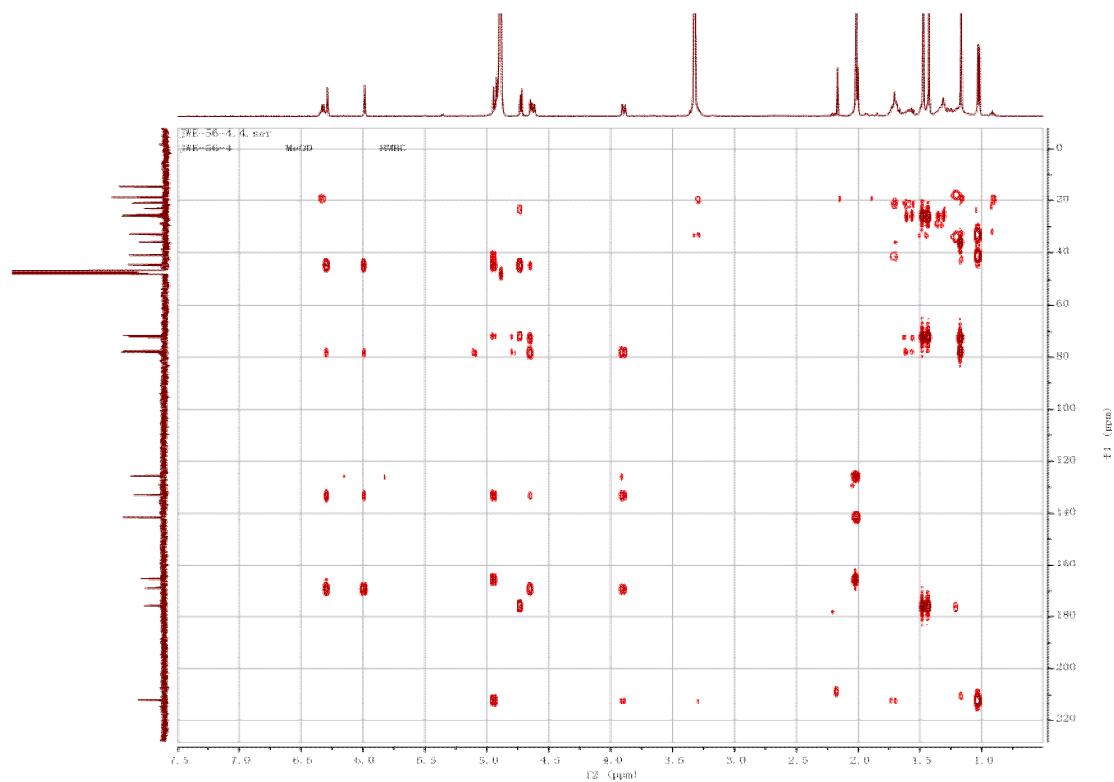


Fig. S3.5 HMBC spectrum (500 MHz) of divarolide B (**3**) in CD_3OD

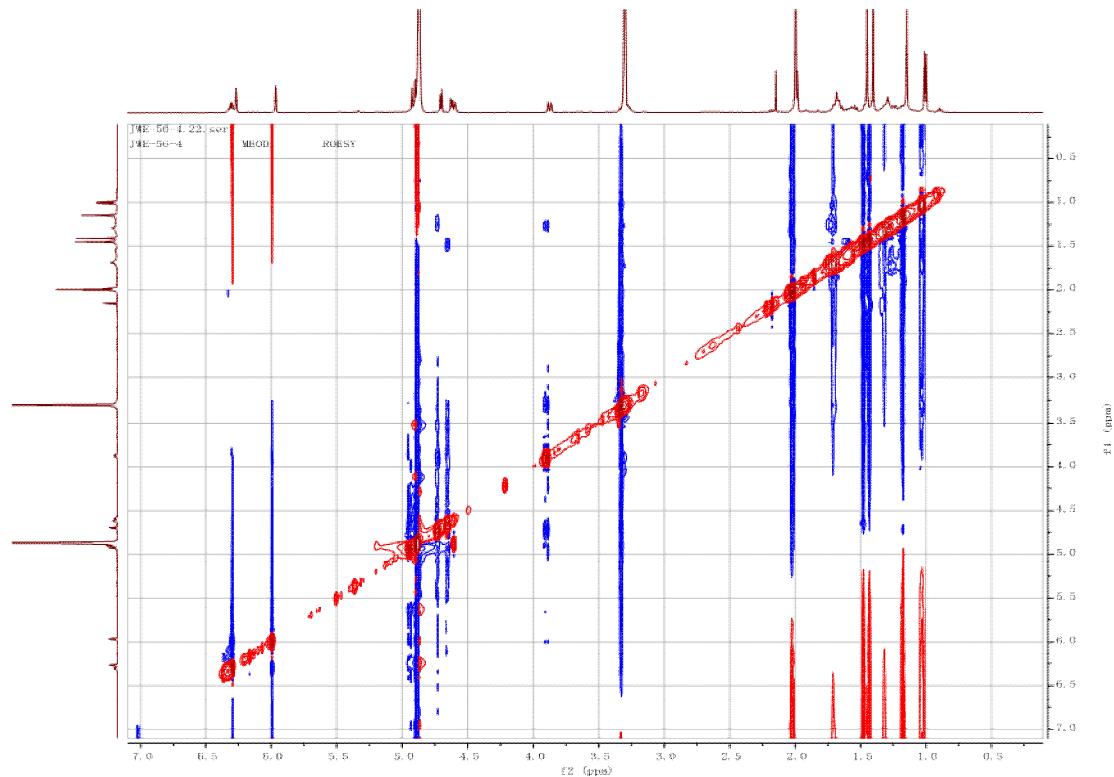
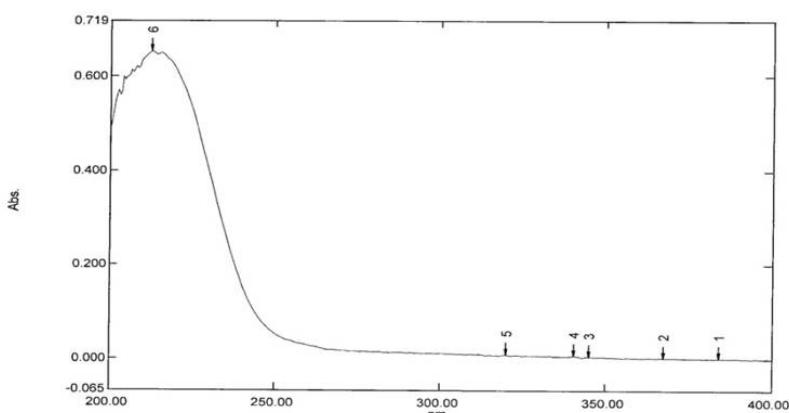


Fig. S3.6 ROESY spectrum (500 MHz) of divarolide B (3) in CD_3OD

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4	(P)	340.50	0.006	
5	(P)	320.00	0.009	
6	(P)	212.50	0.654	

Fig. S3.7 UV spectrum of divarolide B (3)

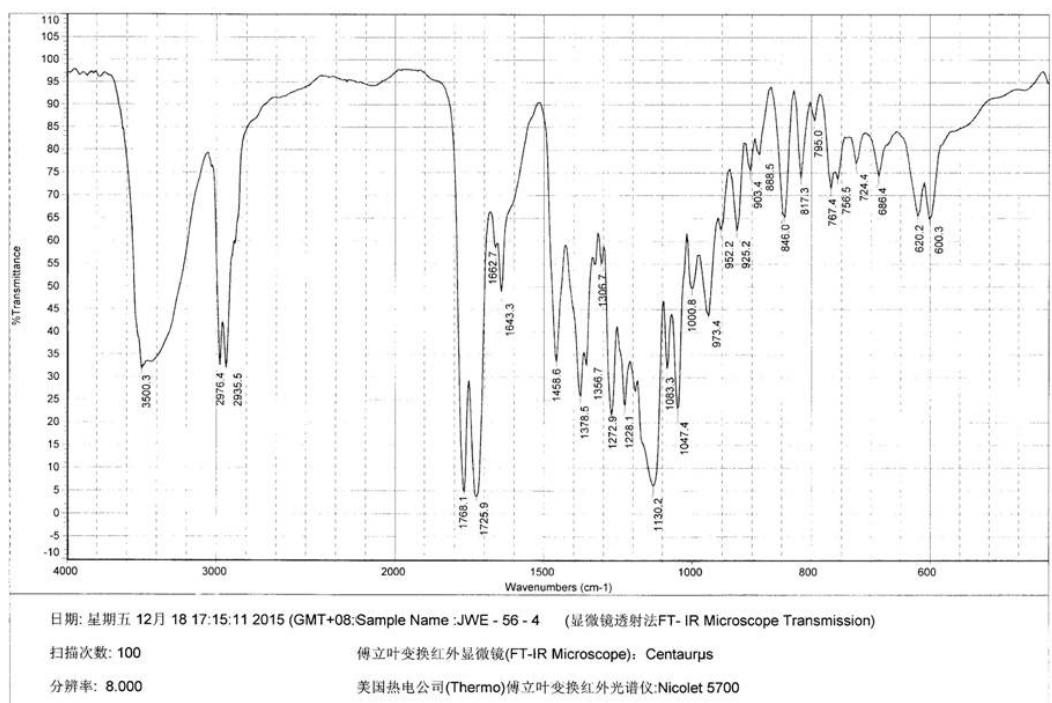


Fig. S3.8 IR spectrum of divarolide B (3)

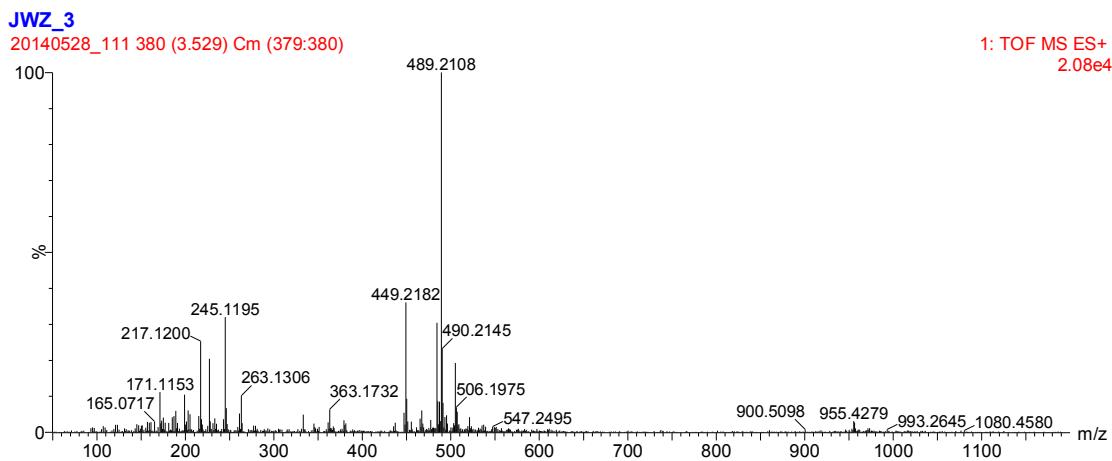


Fig. S3.9 HRESIMS spectrum of divarolide B (3)

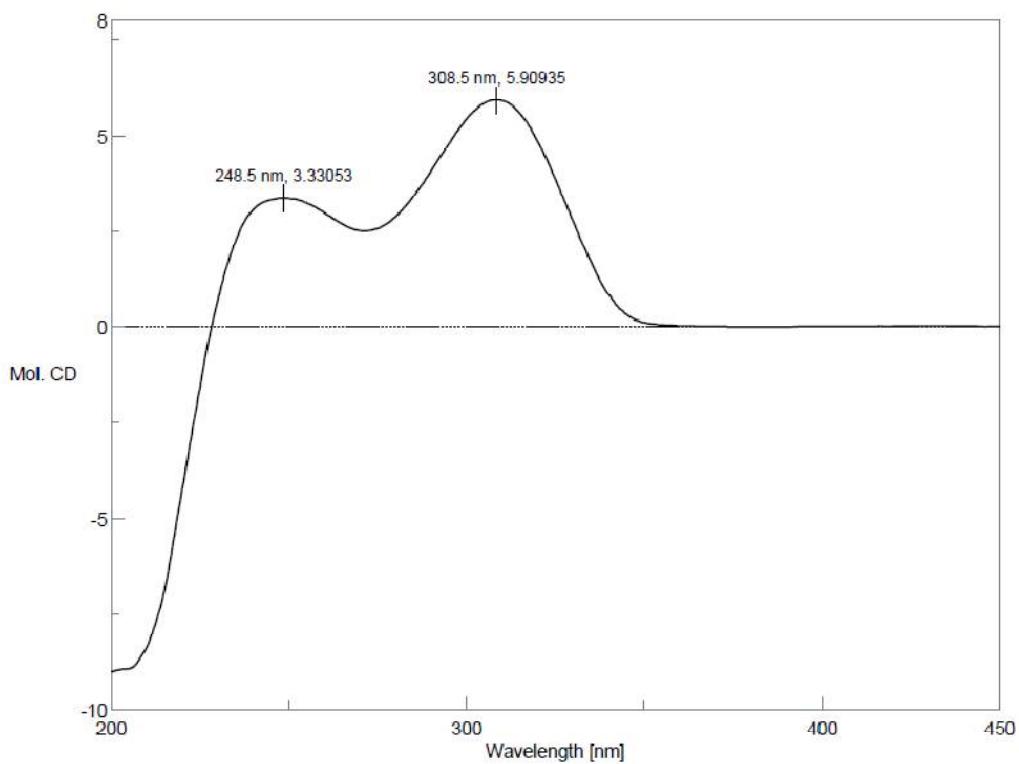


Fig. S3.10 CD spectrum of divarolide B (3)

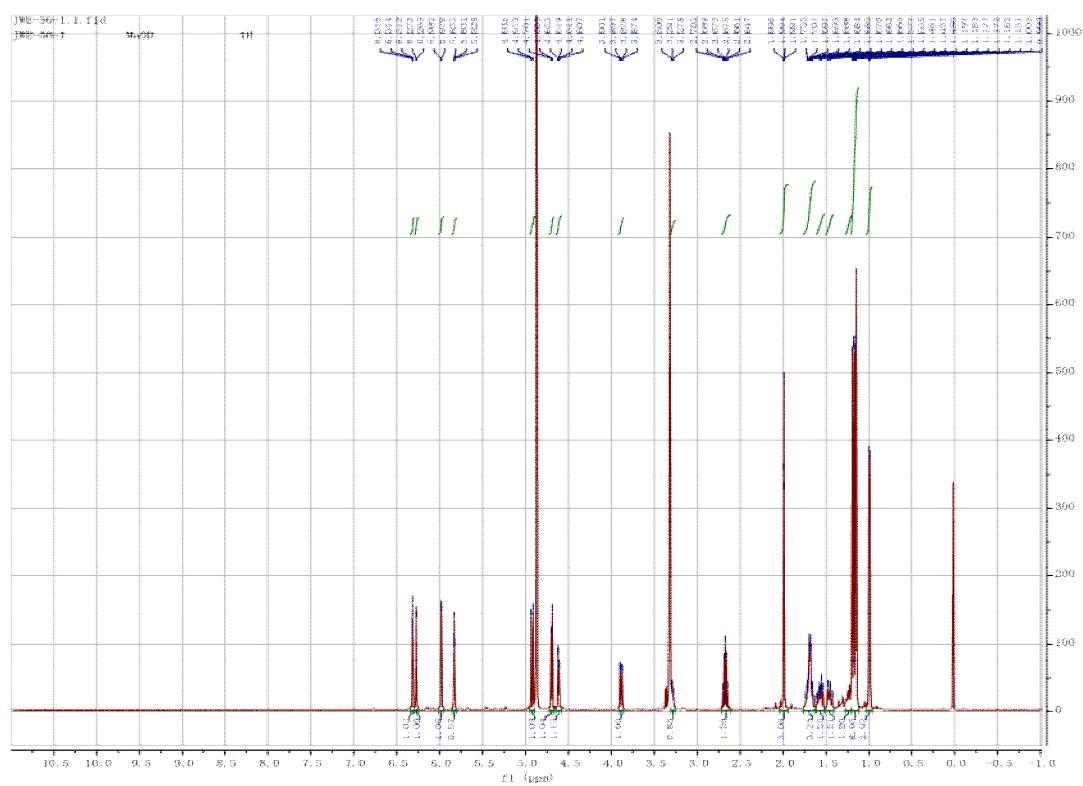


Fig. S4.1 ¹H NMR spectrum (500 MHz) of divarolide C (4) in CD_3OD

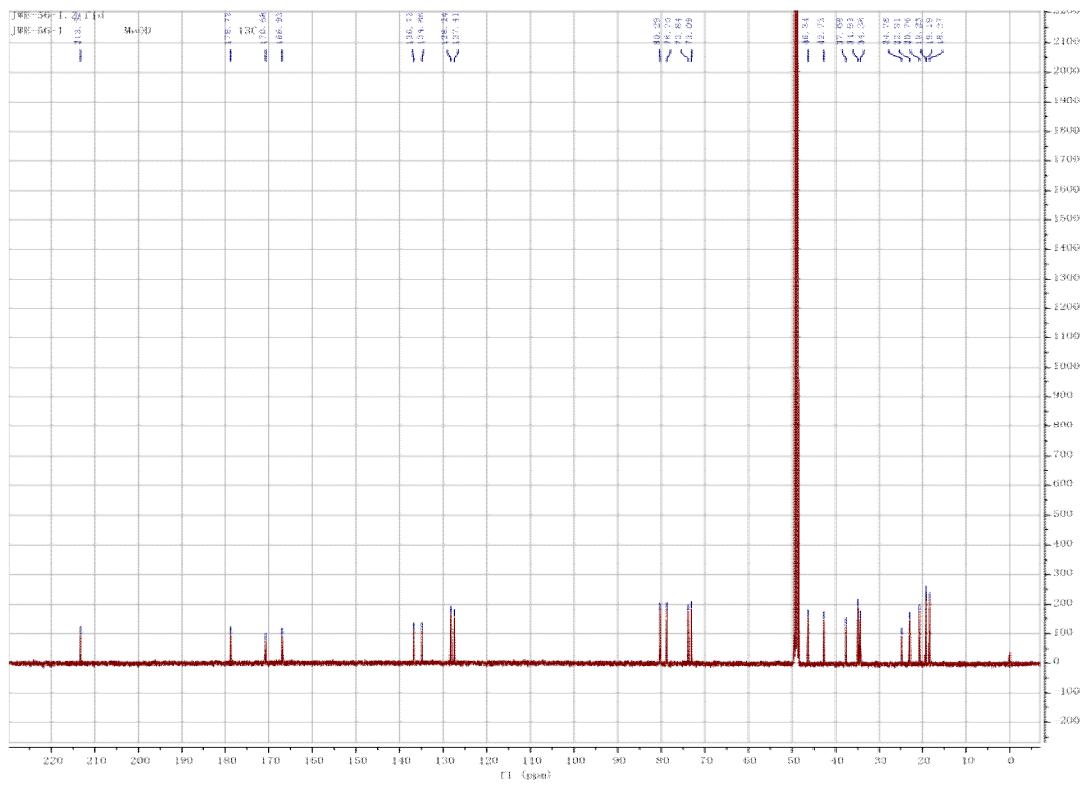


Fig. S4.2 ^{13}C NMR spectrum (500 MHz) of divarolide C (**4**) in CD_3OD

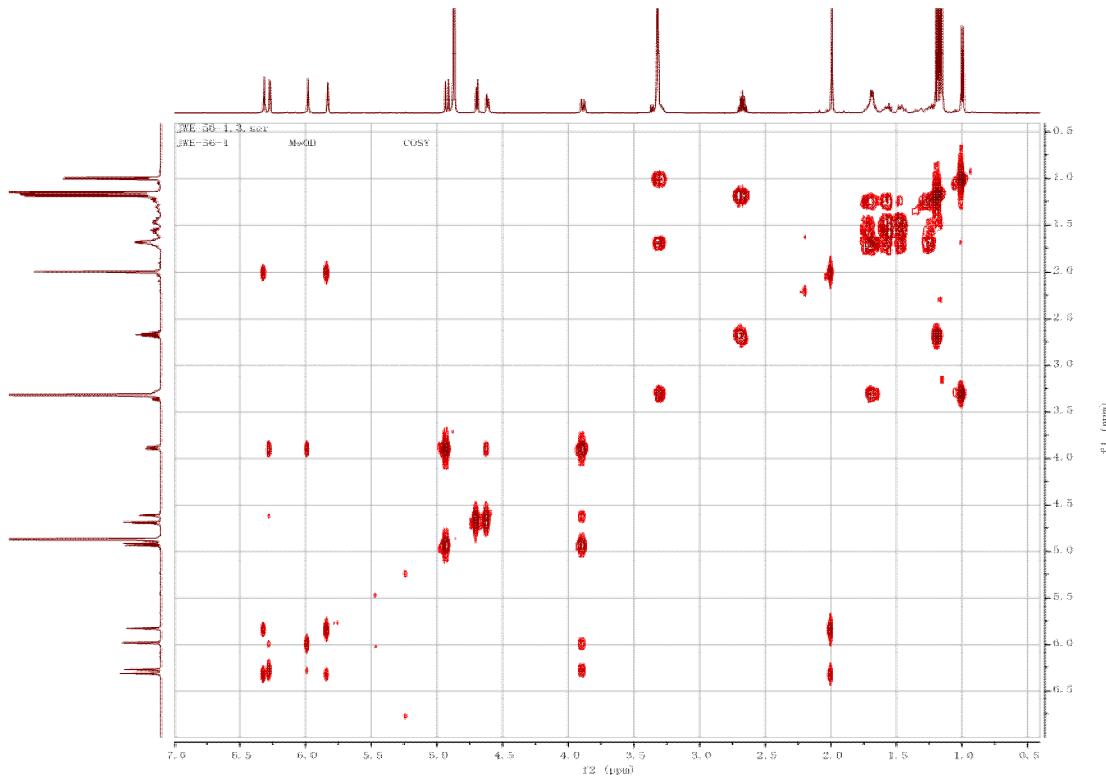


Fig. S4.3 ^1H - ^1H COSY spectrum (500 MHz) of divarolide C (**4**) in CD_3OD

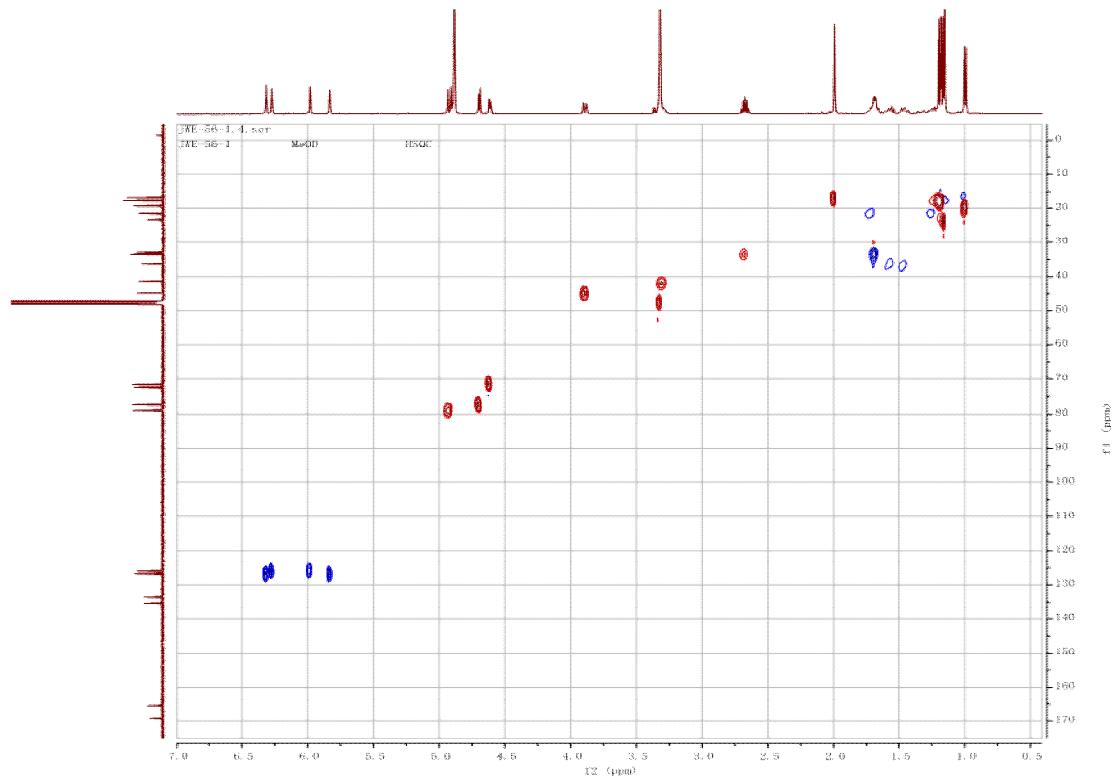


Fig. S4.4 HSQC spectrum (500 MHz) of divarolide C (**4**) in CD_3OD

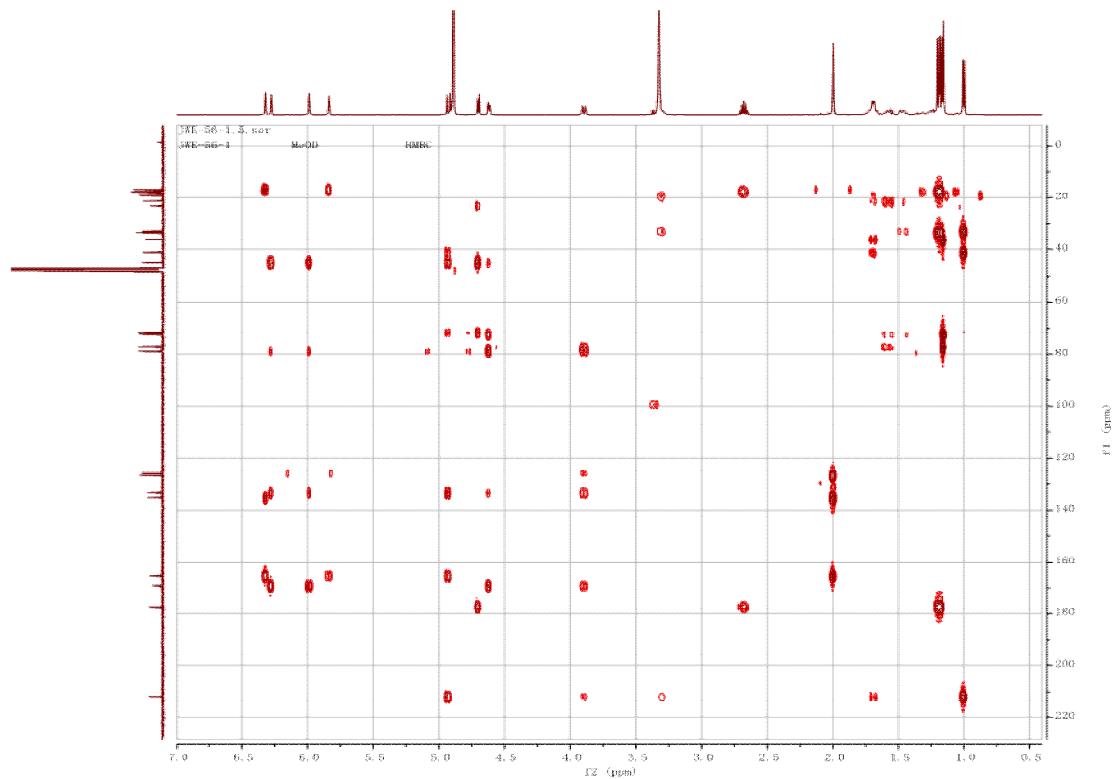


Fig. S4.5 HMBC spectrum (500 MHz) of divarolide C (**4**) in CD_3OD

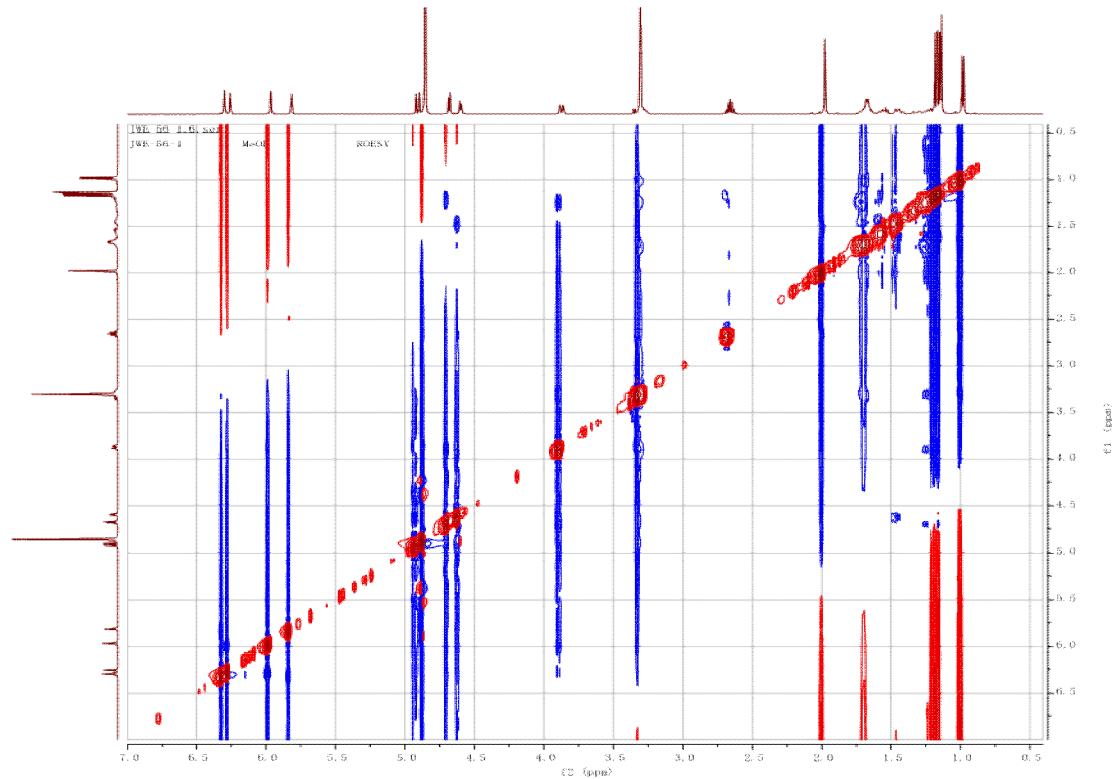


Fig. S4.6 ROESY spectrum (500 MHz) of divarolide C (4) in CD_3OD

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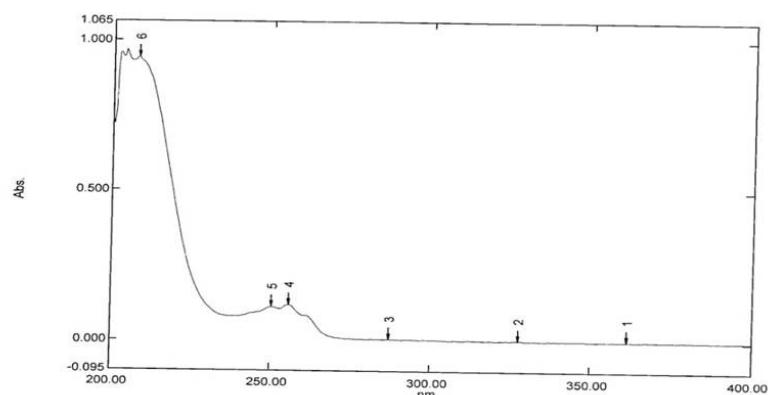


Fig. S4.7 UV spectrum of divarolide C (4)

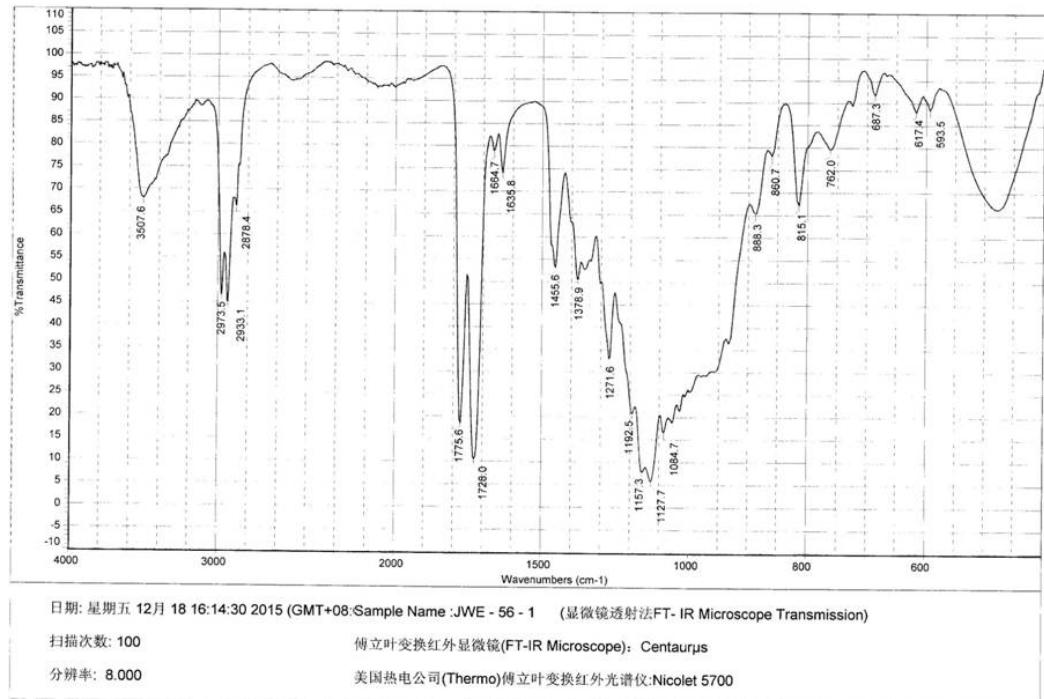


Fig. S4.8 IR spectrum of divarolide C (4)

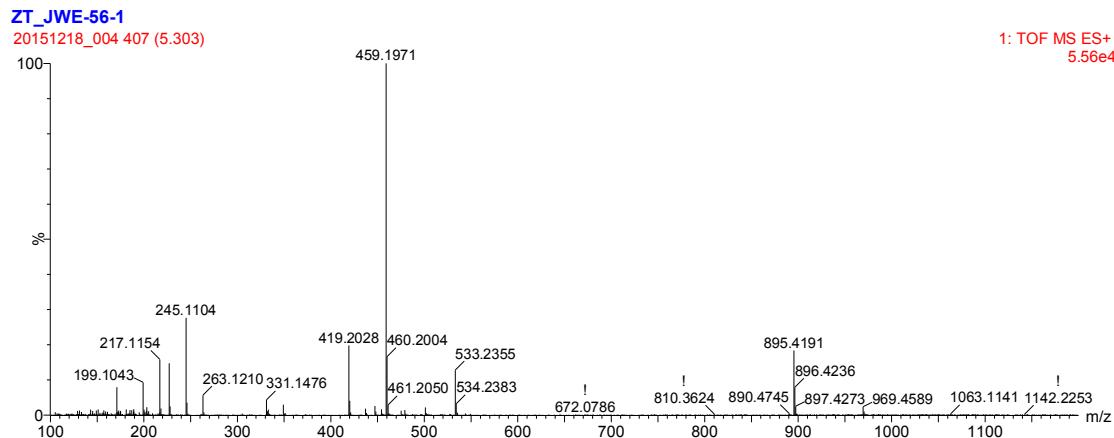


Fig. S4.9 HRESIMS spectrum of divarolide C (4)

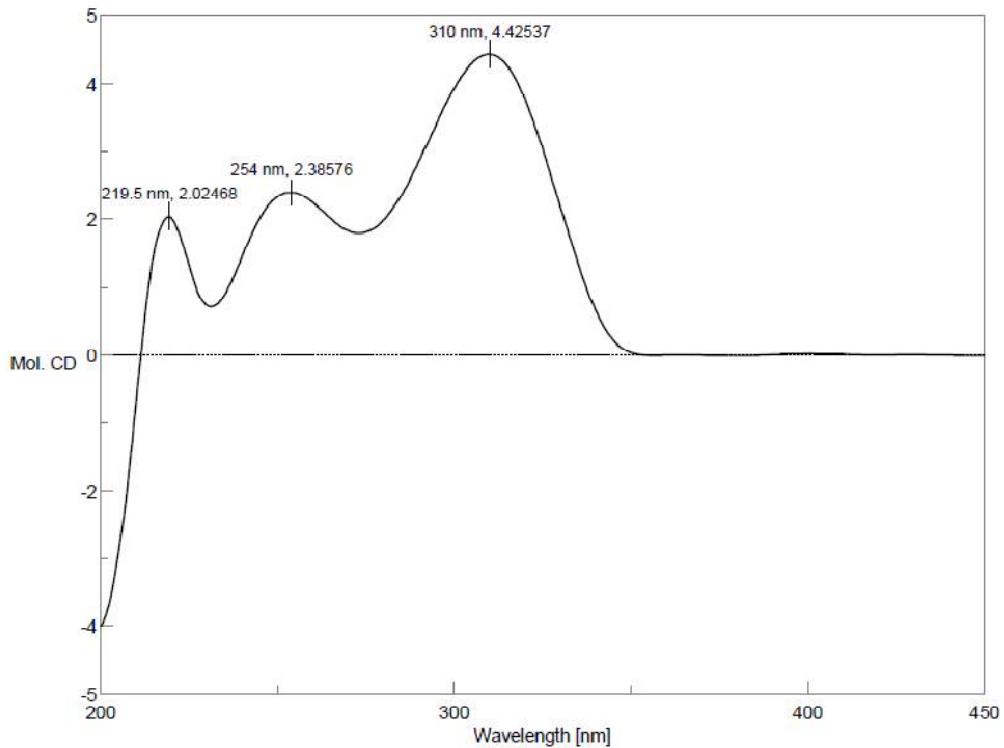


Fig. S4.10 CD spectrum of divarolide C (4)

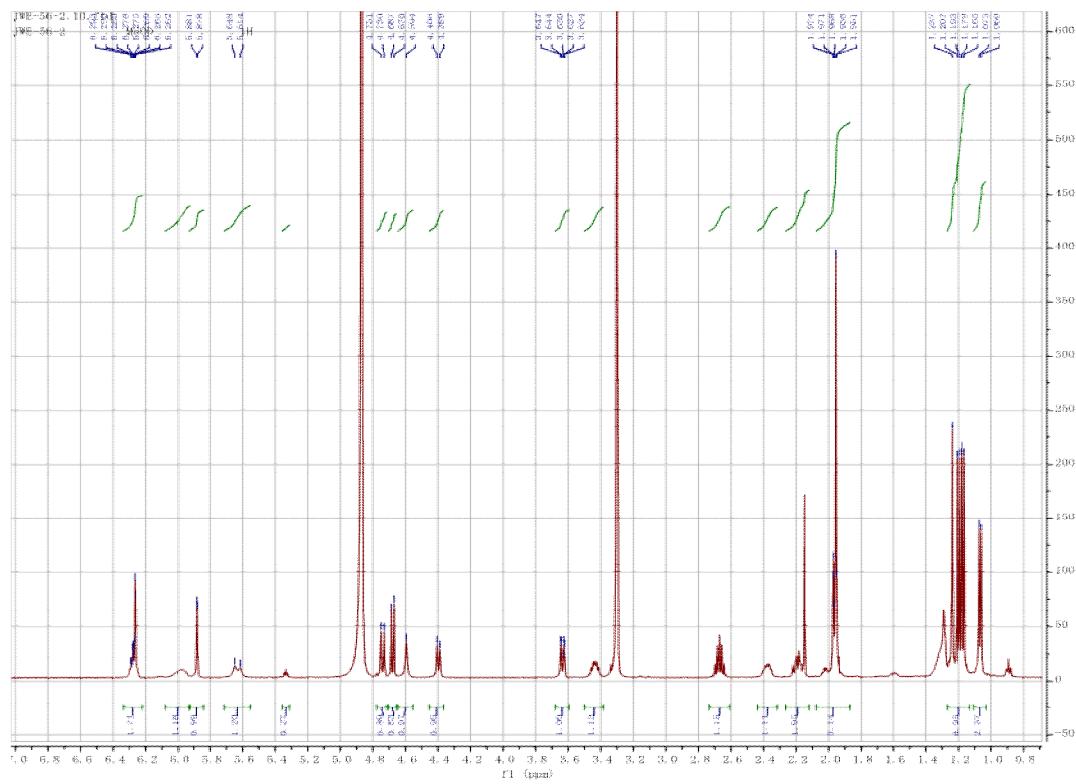


Fig. S5.1 ¹H NMR spectrum (500 MHz) of divarolide D (5) in CD₃OD

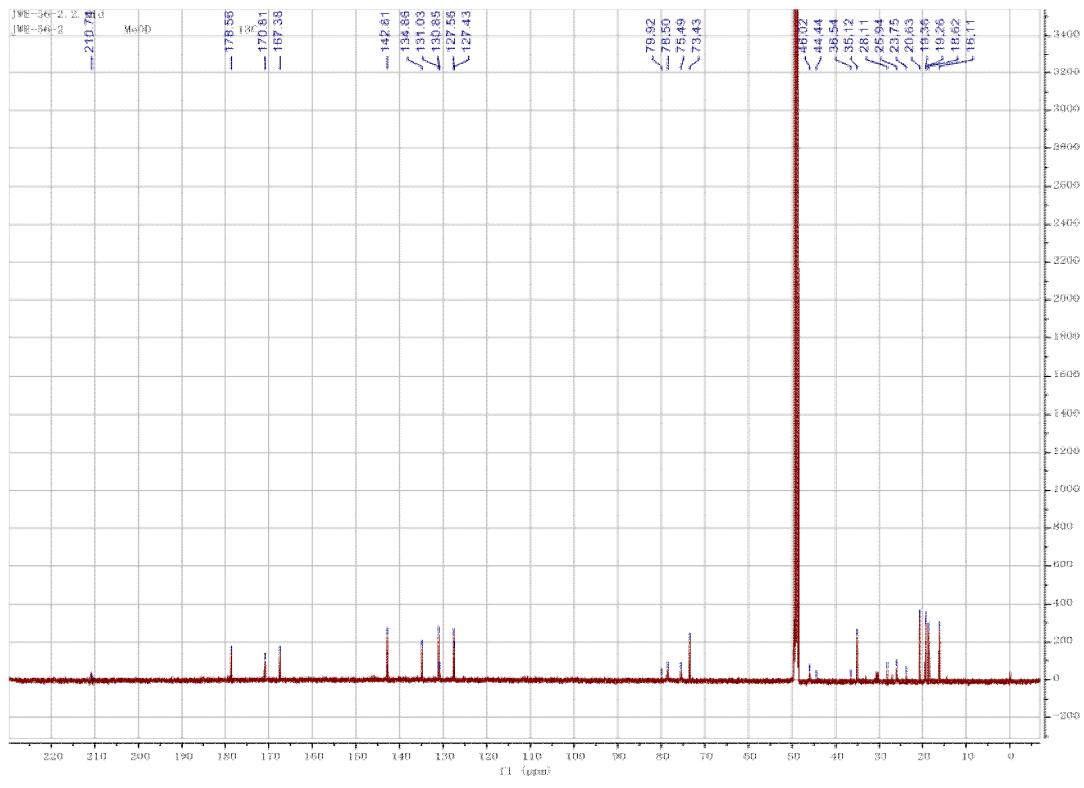


Fig. S5.2 ^{13}C NMR spectrum (500 MHz) of divarolide D (**5**) in CD_3OD

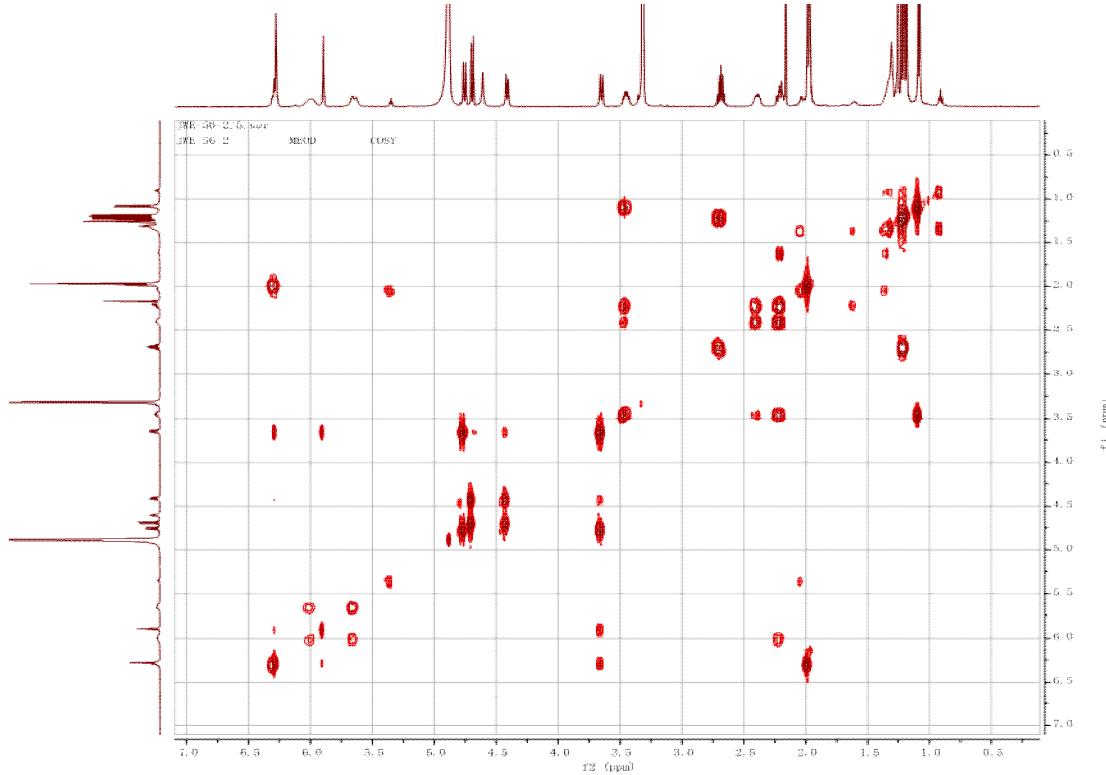


Fig. S5.3 ^1H - ^1H COSY spectrum (500 MHz) of divarolide D (**5**) in CD_3OD

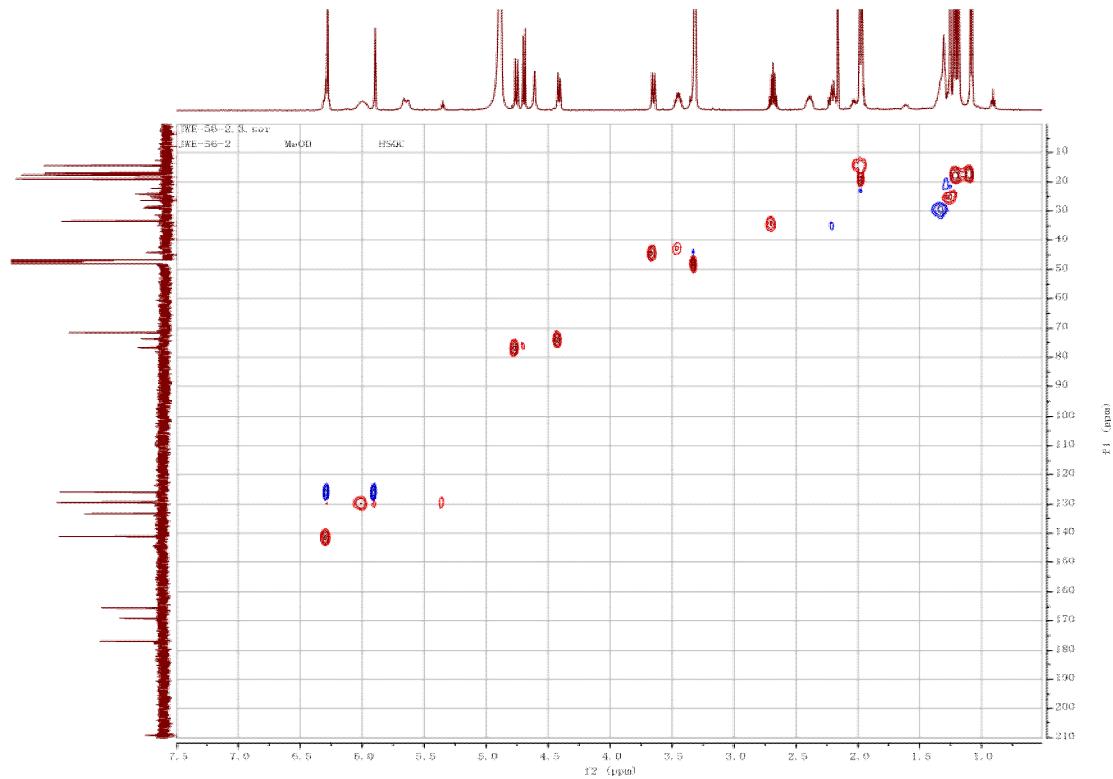


Fig. S5.4 HSQC spectrum (500 MHz) of divarolide D (**5**) in CD_3OD

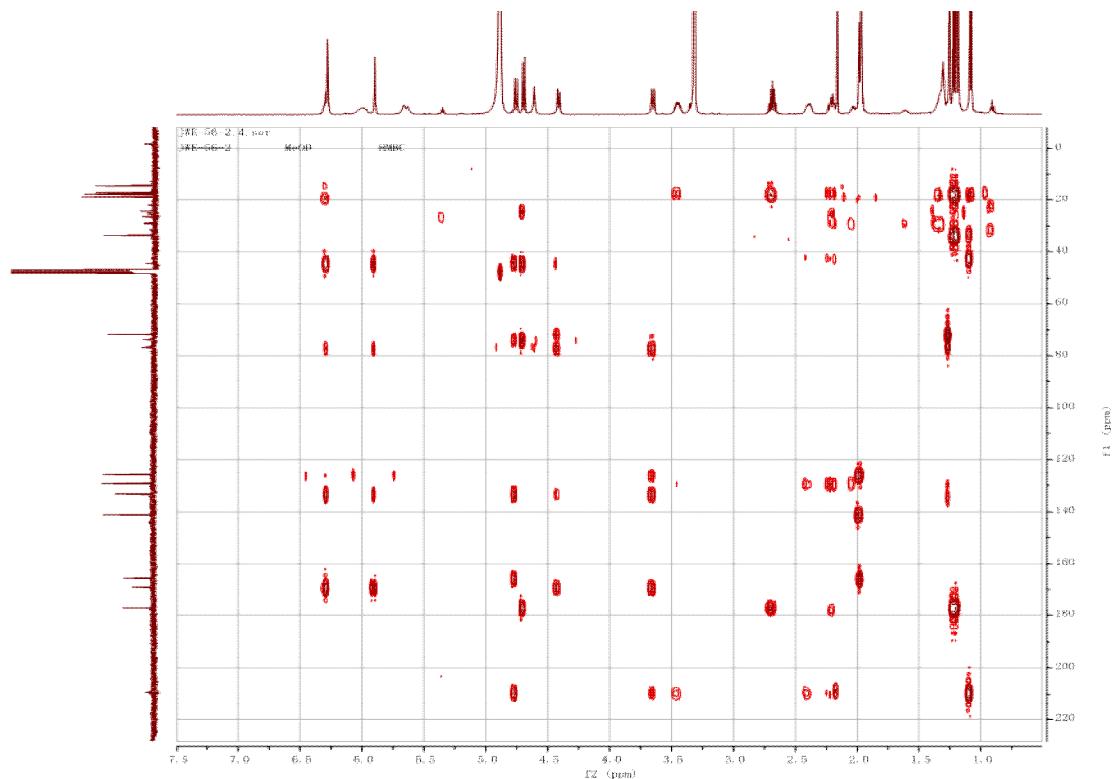


Fig. S5.5 HMBC spectrum (500 MHz) of divarolide D (**5**) in CD_3OD

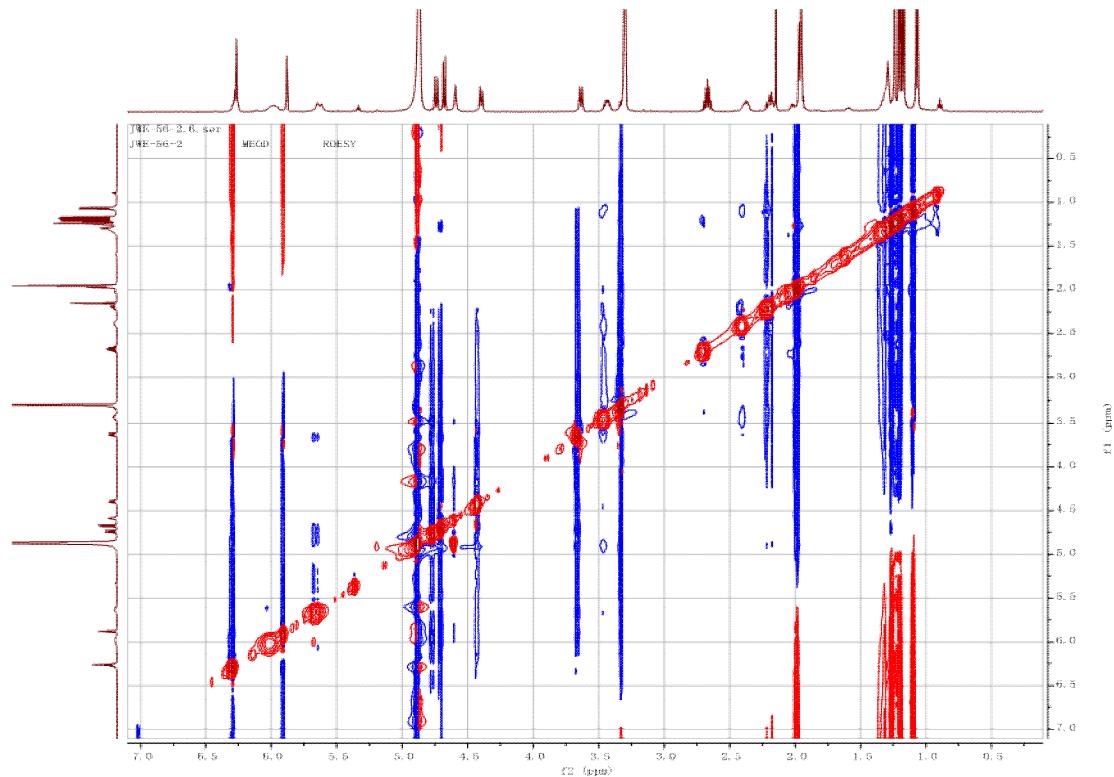
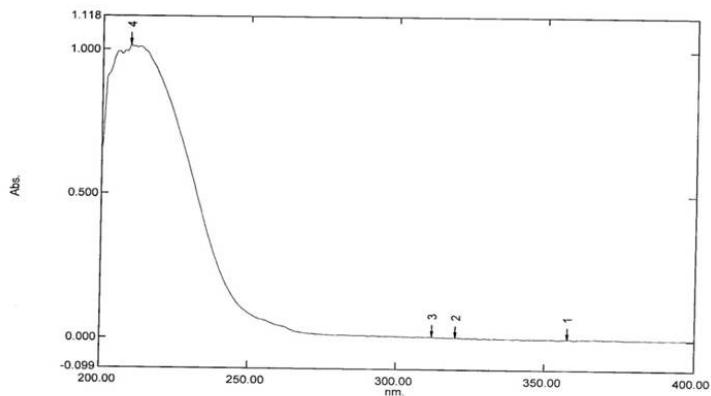


Fig. S5.6 ROESY spectrum (500 MHz) of divarolide D (**5**) in CD₃OD

Auto Print Report 1

2015-12-16 16:09:04

Data Set: Storage 160755 - RawData - D:\2015\JWE-56-2.spc



No.	P/V	Wavelength	Abs.	Description
1	④	357.50	0.005	
2	④	320.00	0.009	
3	④	312.00	0.010	
4	④	209.50	1.017	

Fig. S5.7 UV spectrum of divarolide D (**5**)

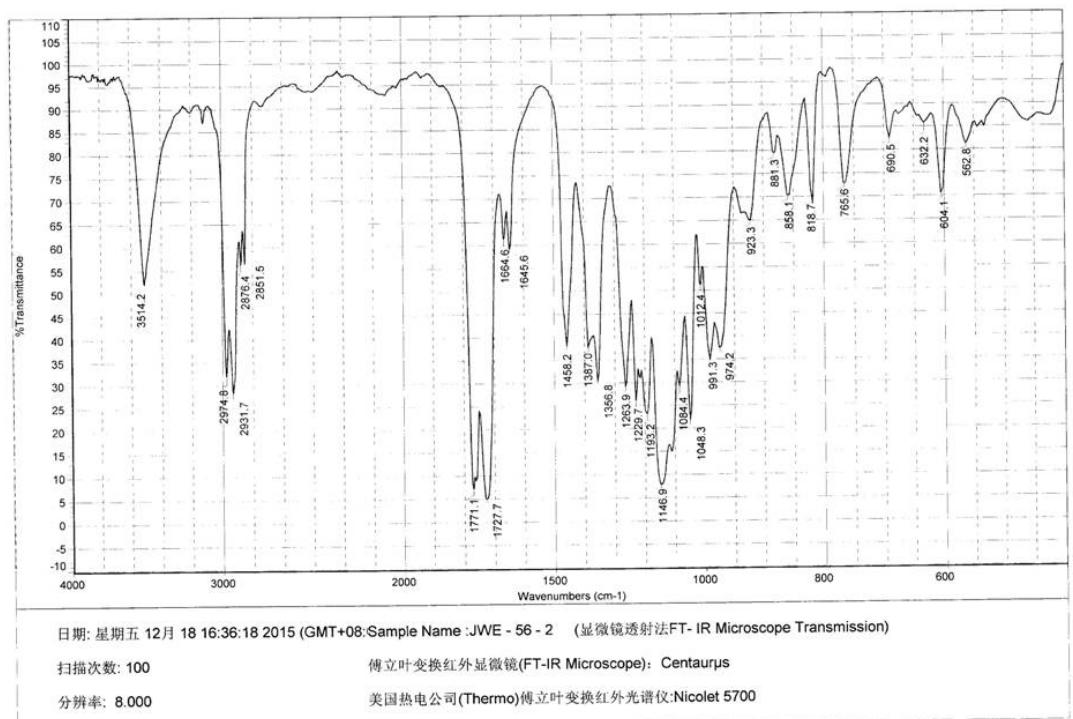


Fig. S5.8 IR spectrum of divarolide D (5)

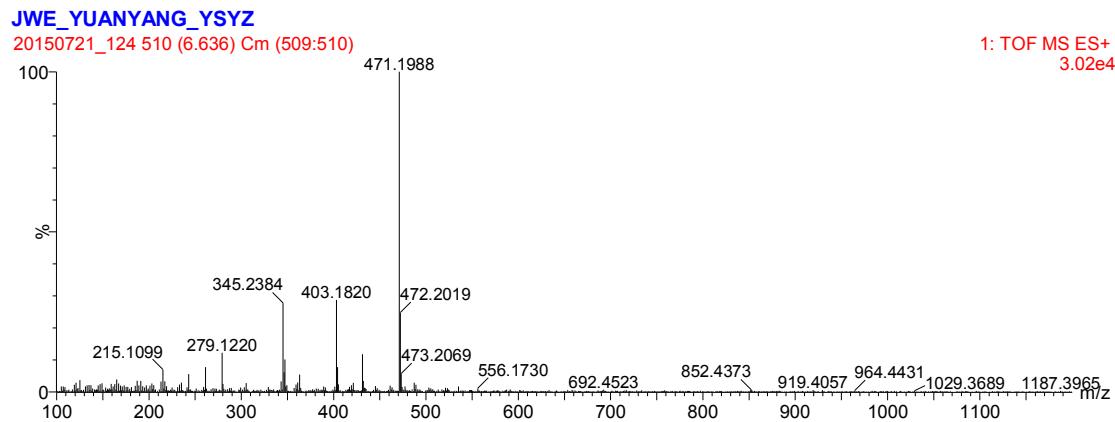


Fig. S5.9 HRESIMS spectrum of divarolide D (5)

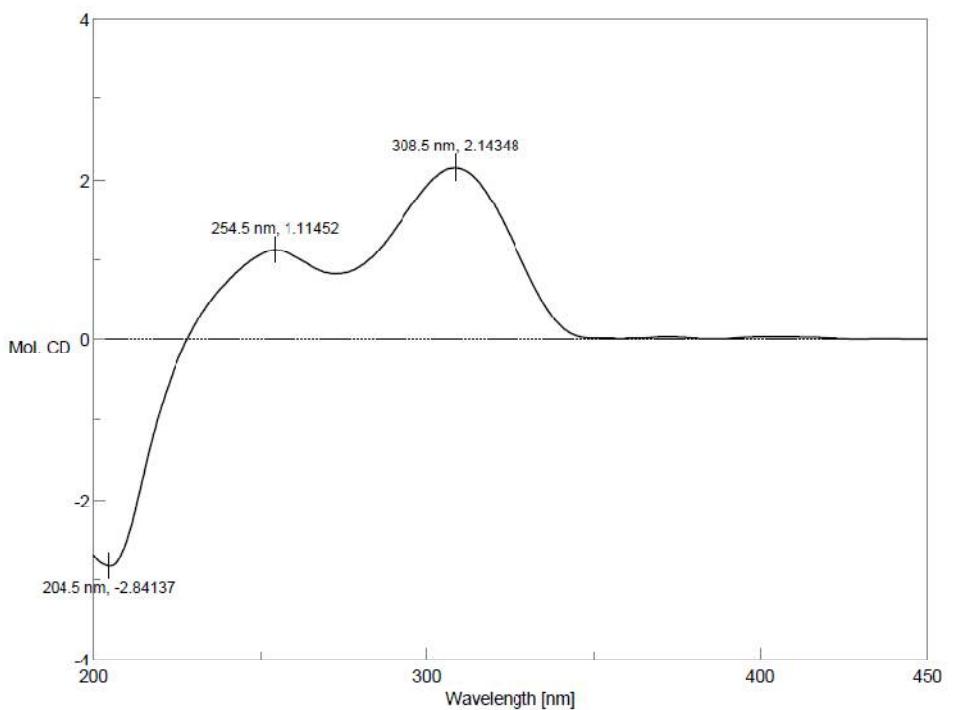


Fig. S5.10 CD spectrum of divarolide D (**5**)

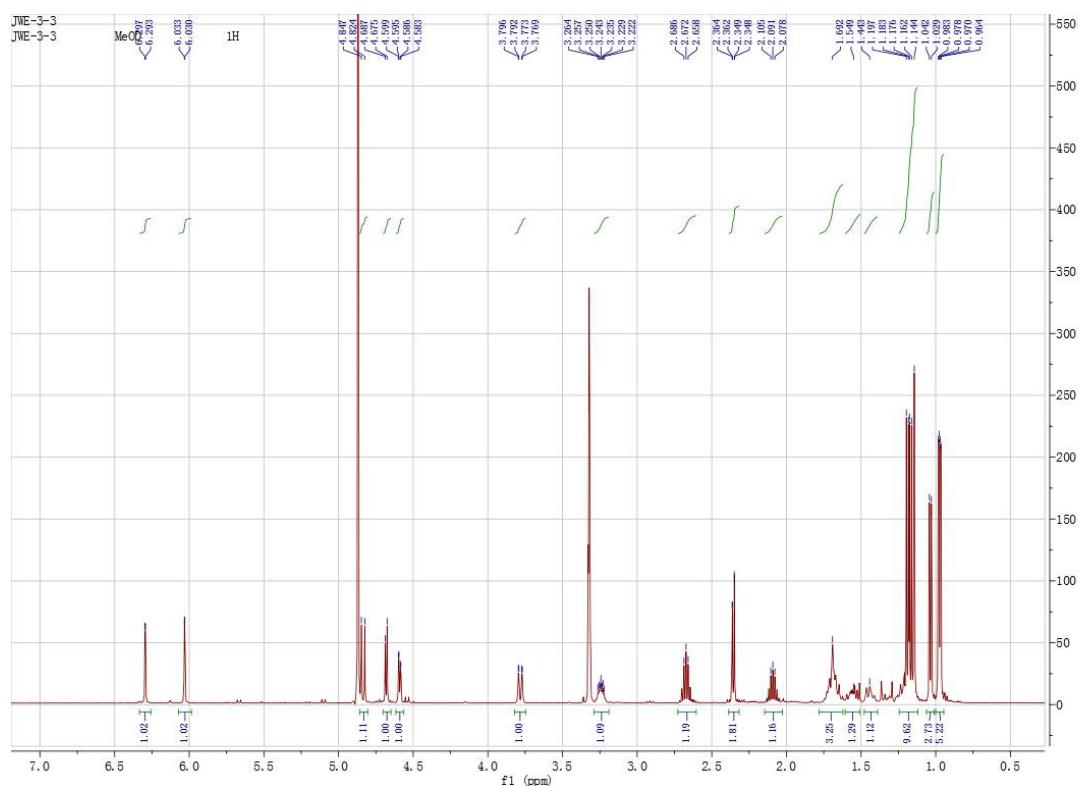


Fig. S6.1 ^1H NMR spectrum (500 MHz) of incaspitolide B₁ (**6**) in CD₃OD

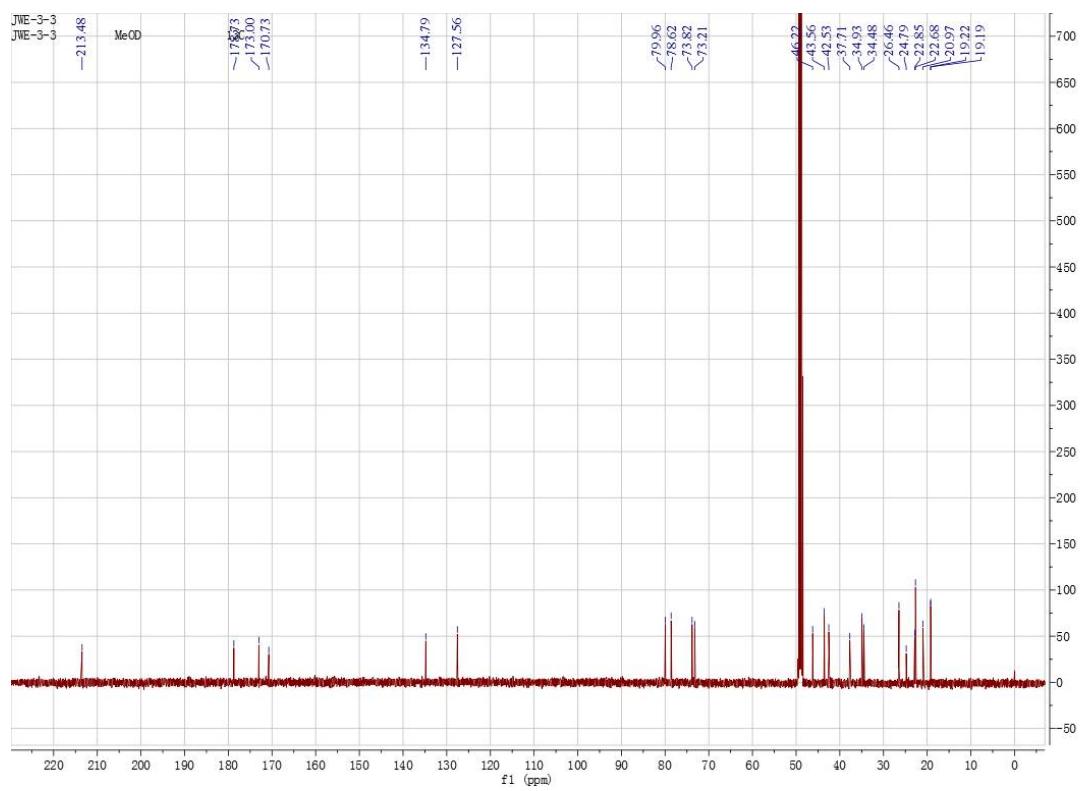


Fig. S6.2 ^{13}C NMR spectrum (500 MHz) of incaspitolide B₁ (**6**) in CD₃OD

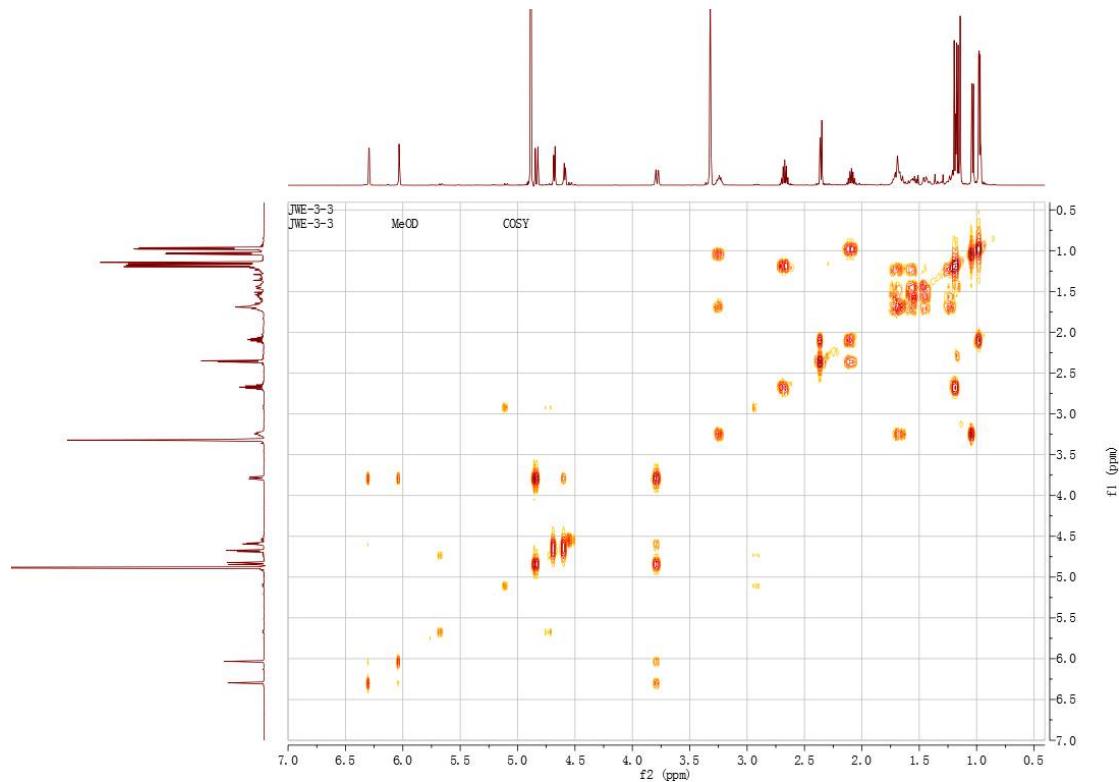


Fig. S6.3 ^1H - ^1H COSY spectrum (500 MHz) of incaspitolide B₁ (**6**) in CD₃OD

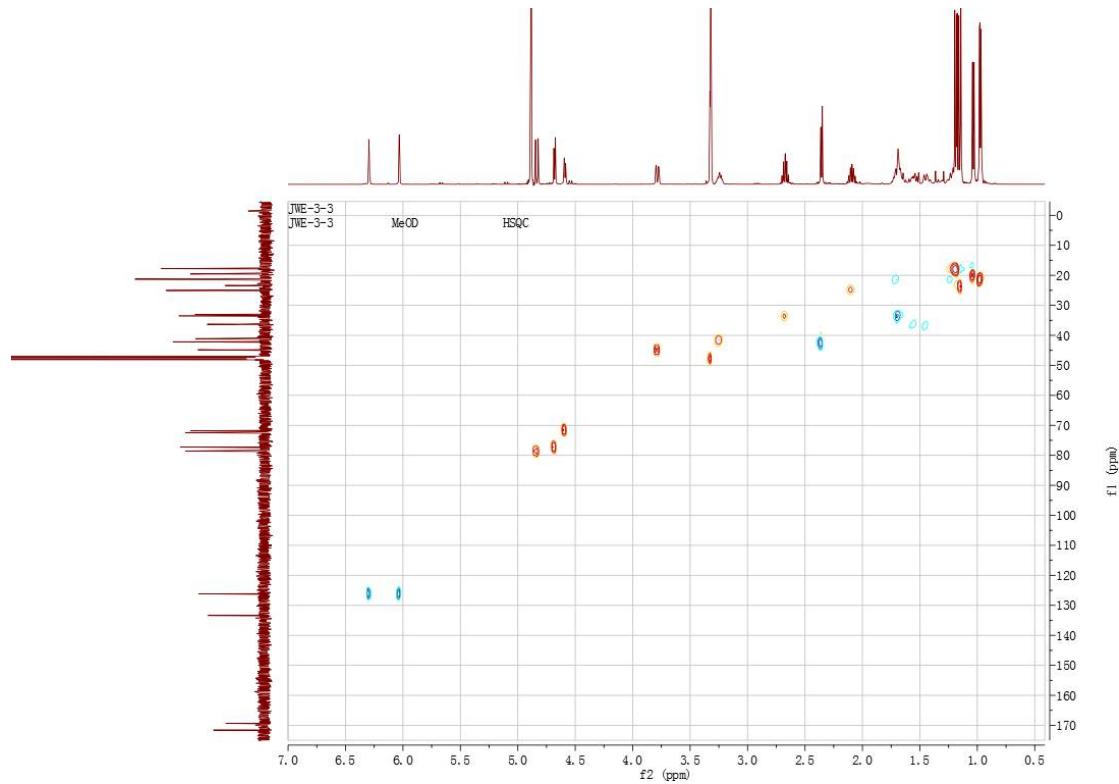


Fig. S6.4 HSQC spectrum (500 MHz) of incaspitolide B₁ (**6**) in CD₃OD

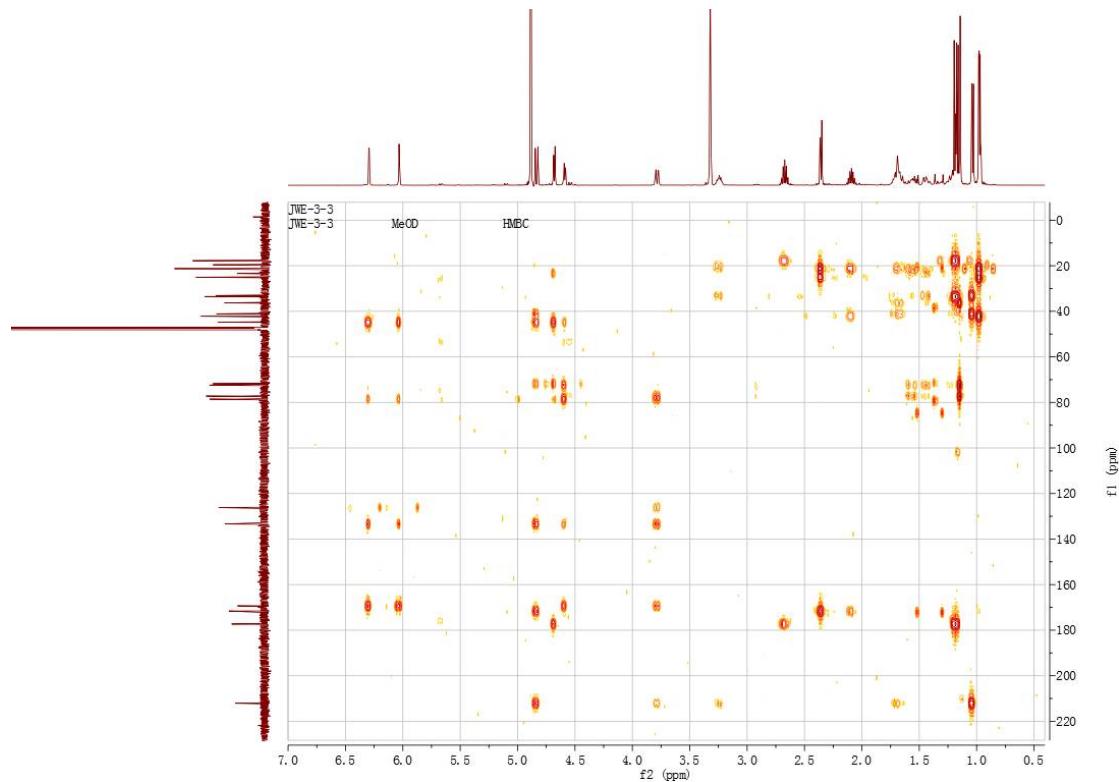


Fig. S6.5 HMBC spectrum (500 MHz) of incaspitolide B₁ (**6**) in CD₃OD

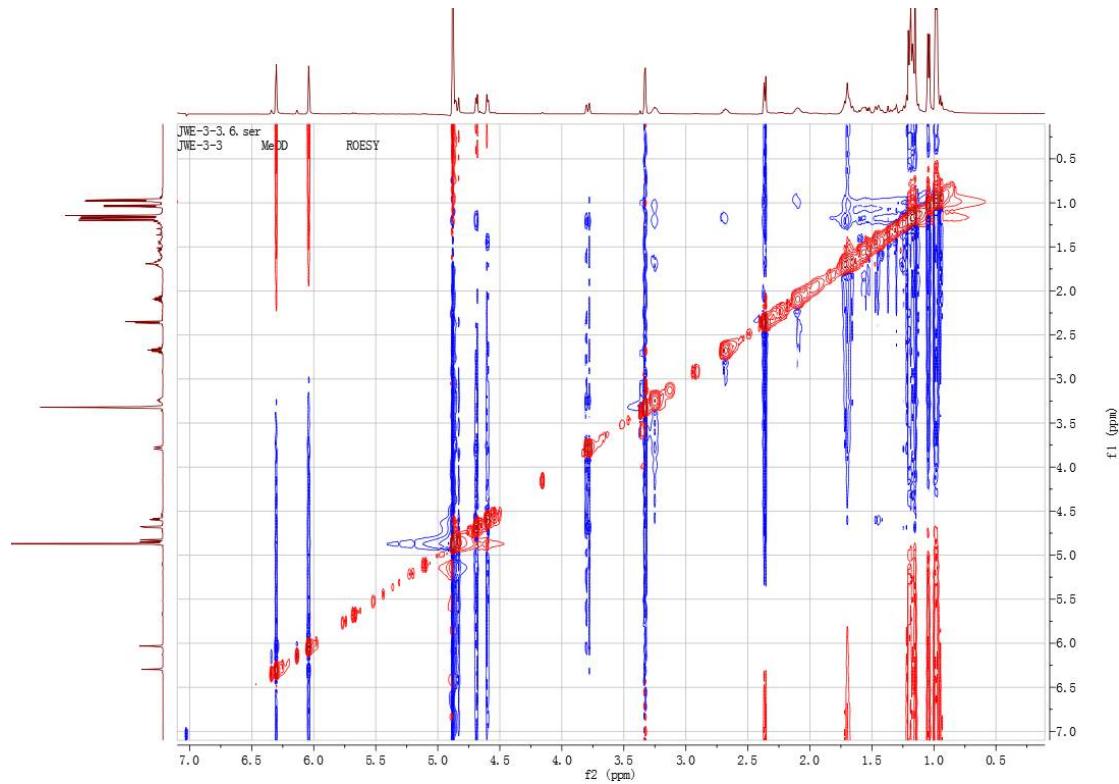
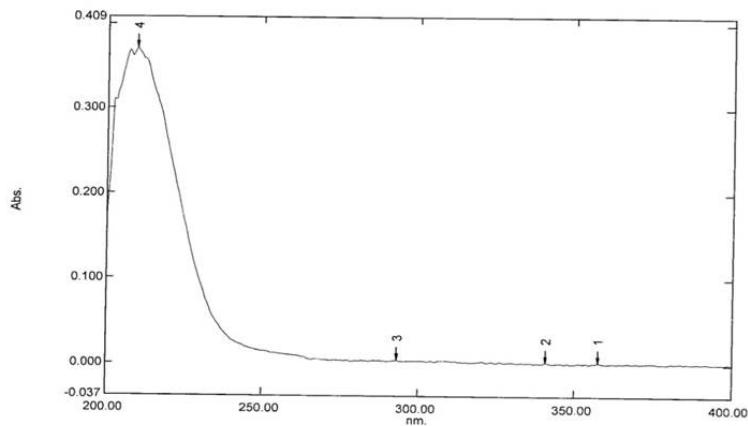


Fig. S6.6 ROESY spectrum (500 MHz) of incasitolide B₁ (**6**) in CD₃OD

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Data Set: Storage 162036 - RawData - D:\2015\JWE-3-3.spc



No.	P/V	Wavelength	Abs.	Description
1	④	357.50	0.002	
2	④	341.00	0.002	
3	④	293.00	0.004	
4	④	209.50	0.371	

Fig. S6.7 UV spectrum of incasitolide B₁ (**6**)

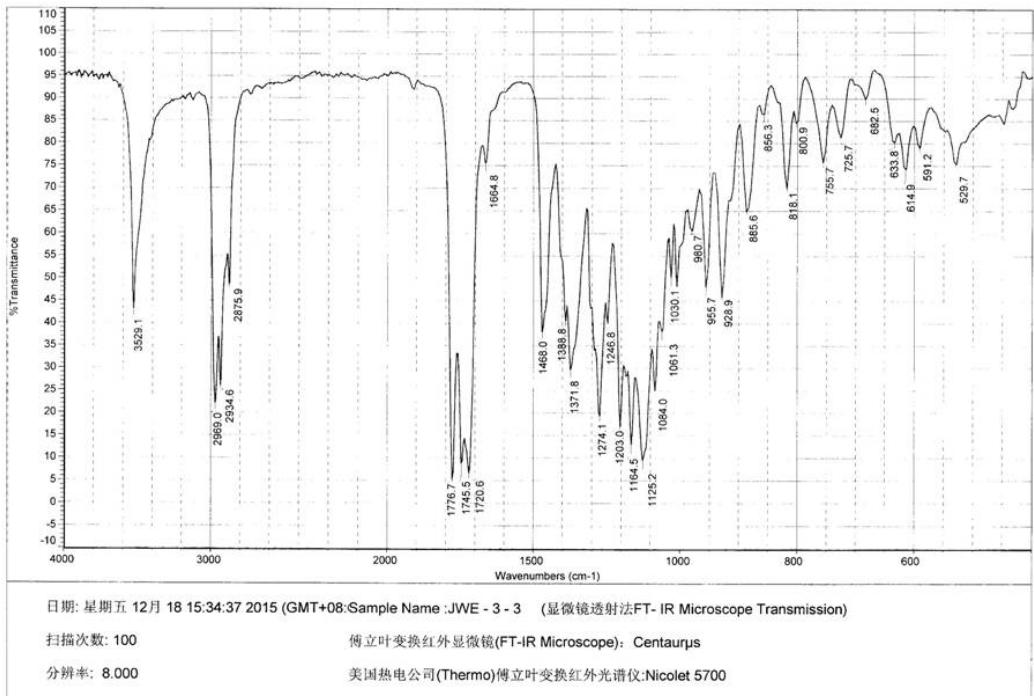


Fig. S6.8 IR spectrum of incaspitolide B₁ (6)

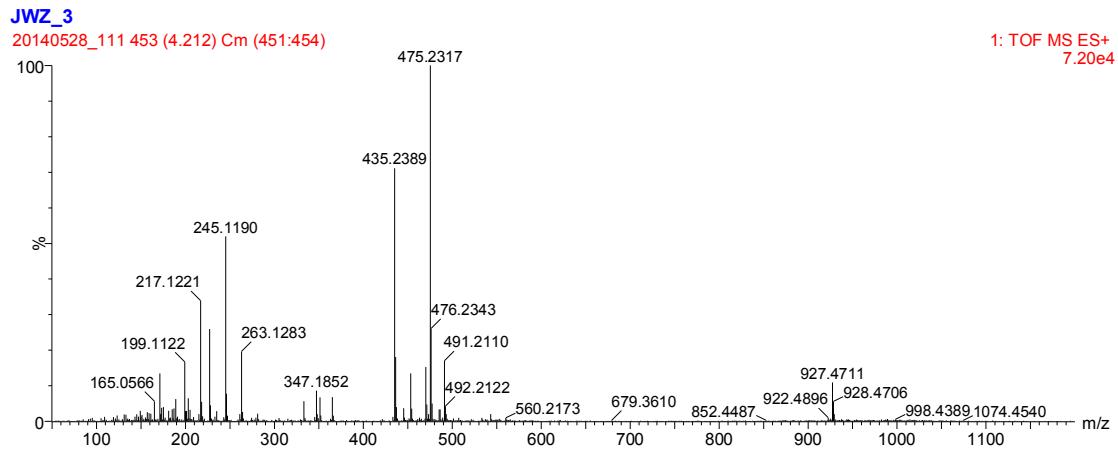


Fig. S6.9 HRESIMS spectrum of incaspitolide B₁ (6)

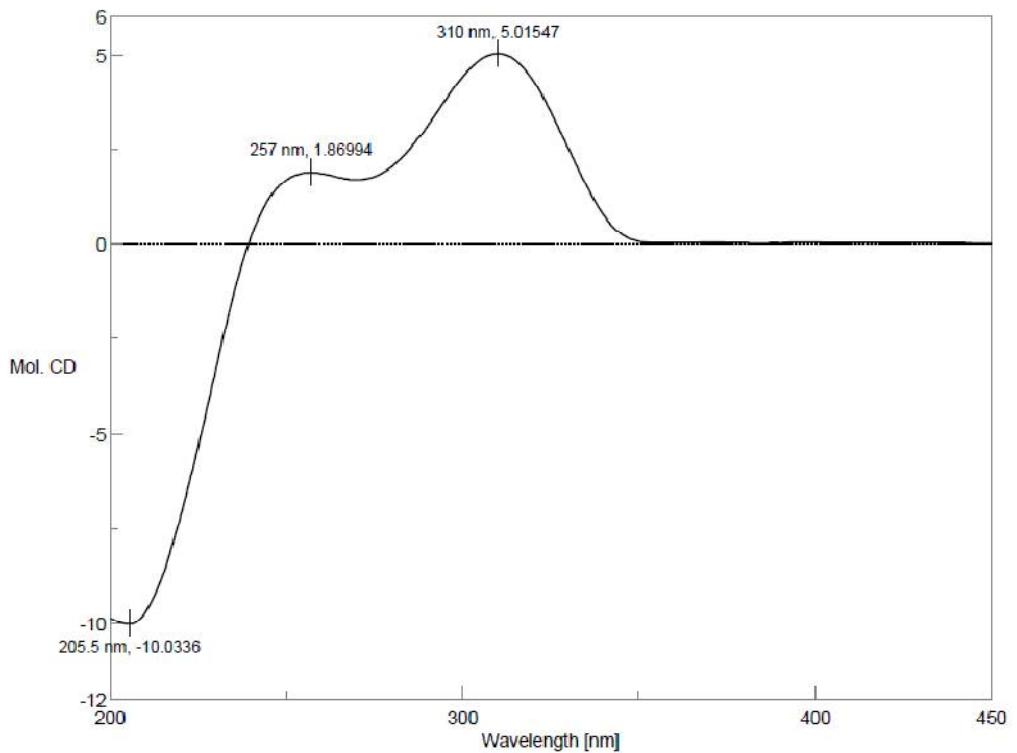


Fig. S6.10 CD spectrum of incaspitolide B₁ (**6**)

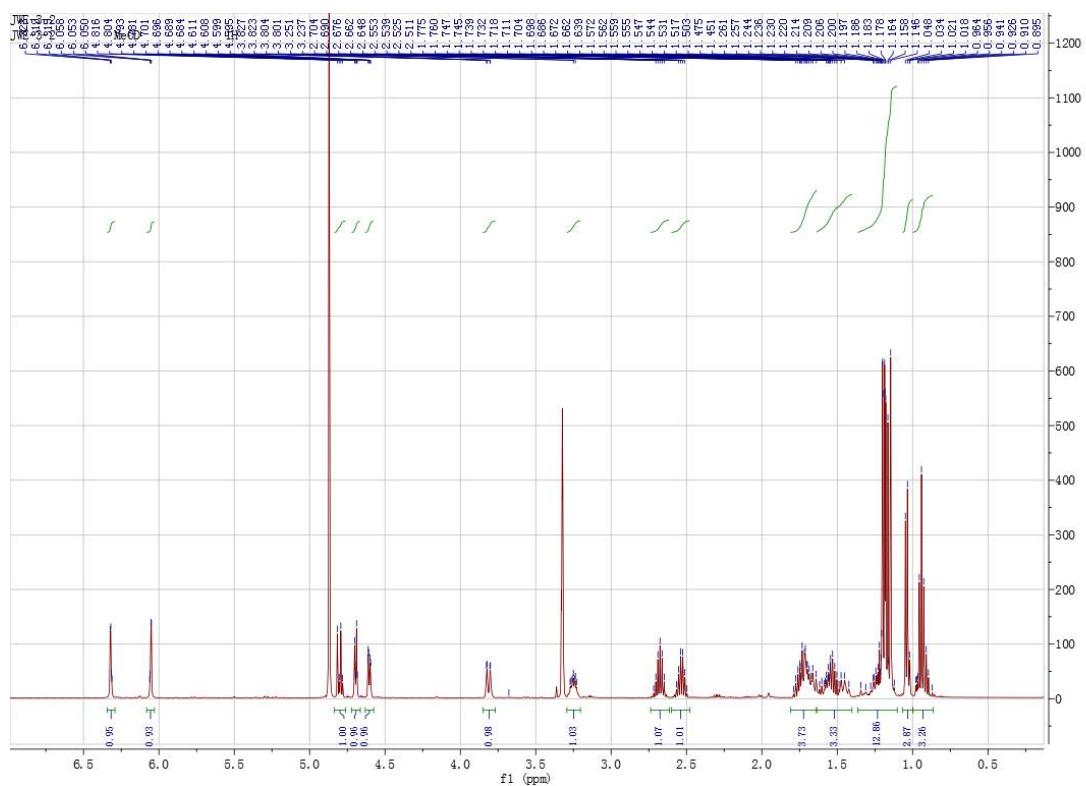


Fig. S7.1 ^1H NMR spectrum (500 MHz) of incaspitolide B₂ (**7**) in CD₃OD

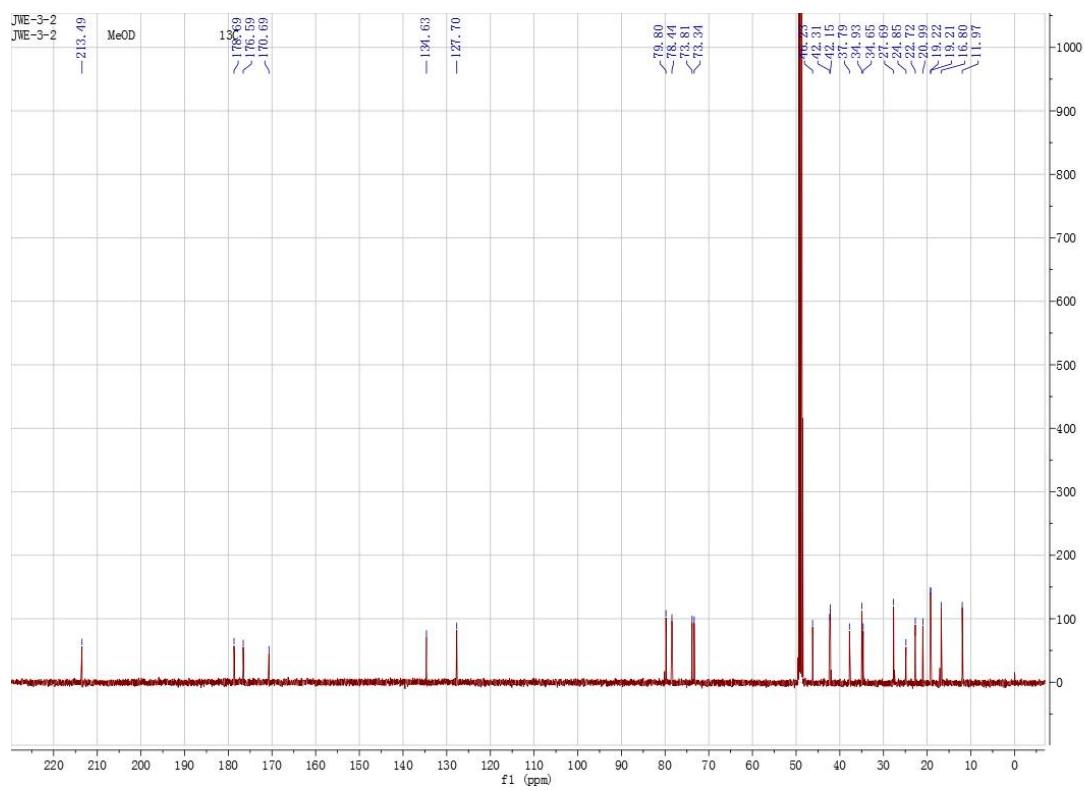


Fig. S7.2 ^{13}C NMR spectrum (500 MHz) of incaspitolide B₂ (**7**) in CD_3OD

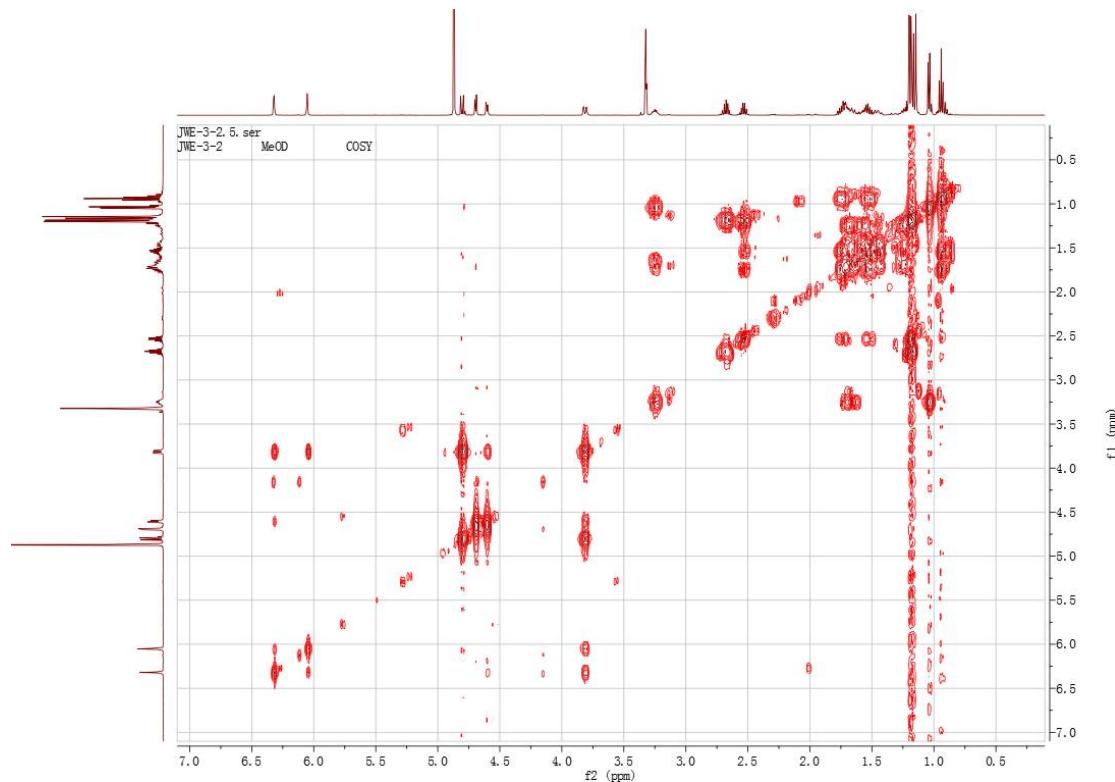


Fig. S7.3 ^1H - ^1H COSY spectrum (500 MHz) of incaspitolide B₂ (**7**) in CD_3OD

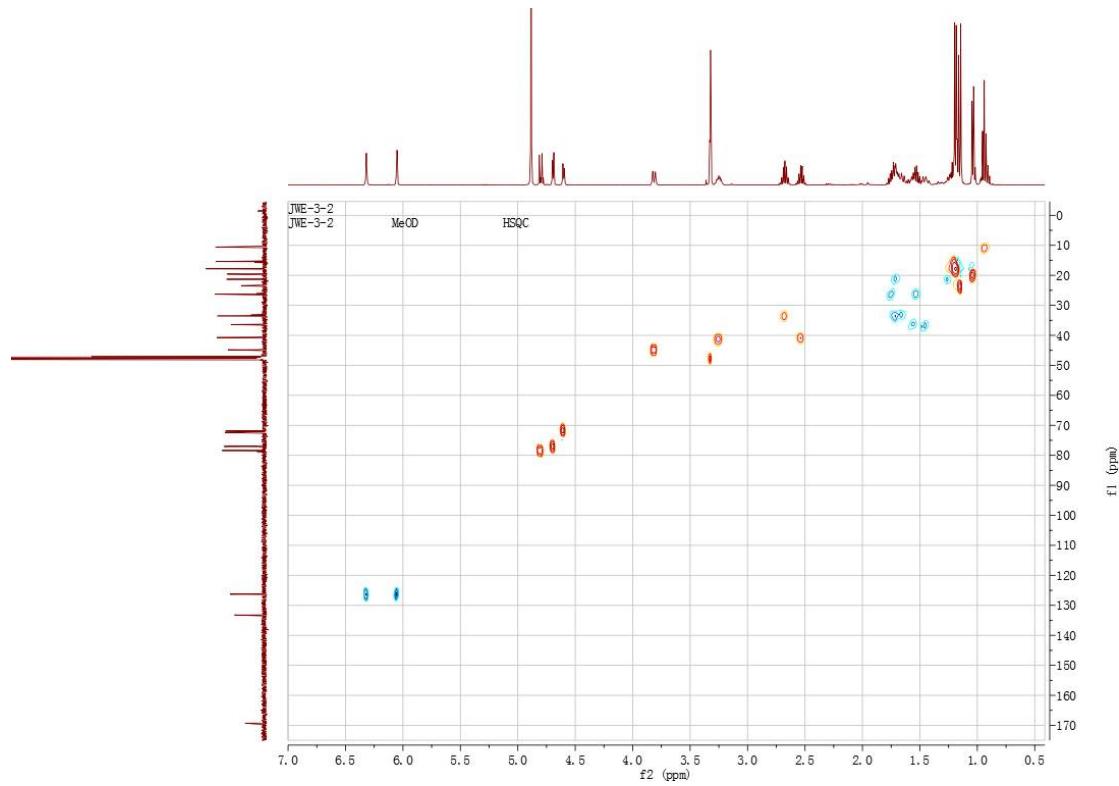


Fig. S7.4 HSQC spectrum (500 MHz) of incaspitolide B₂ (**7**) in CD₃OD

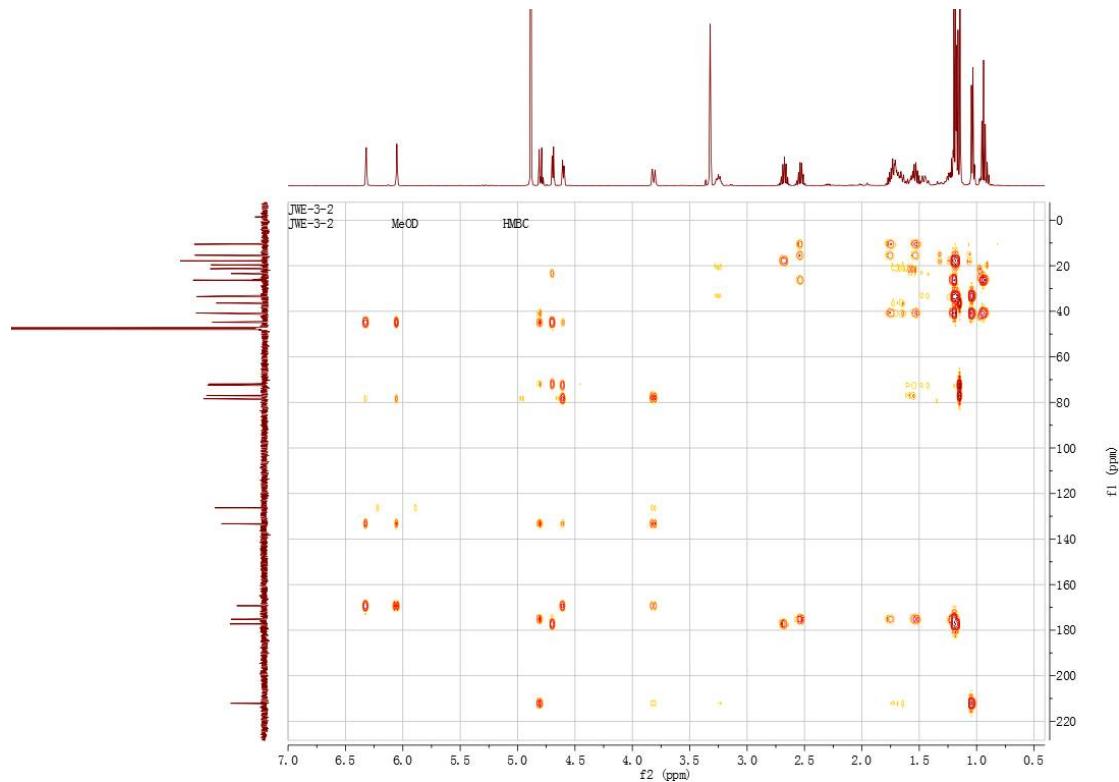


Fig. S7.5 HMBC spectrum (500 MHz) of incaspitolide B₂ (**7**) in CD₃OD

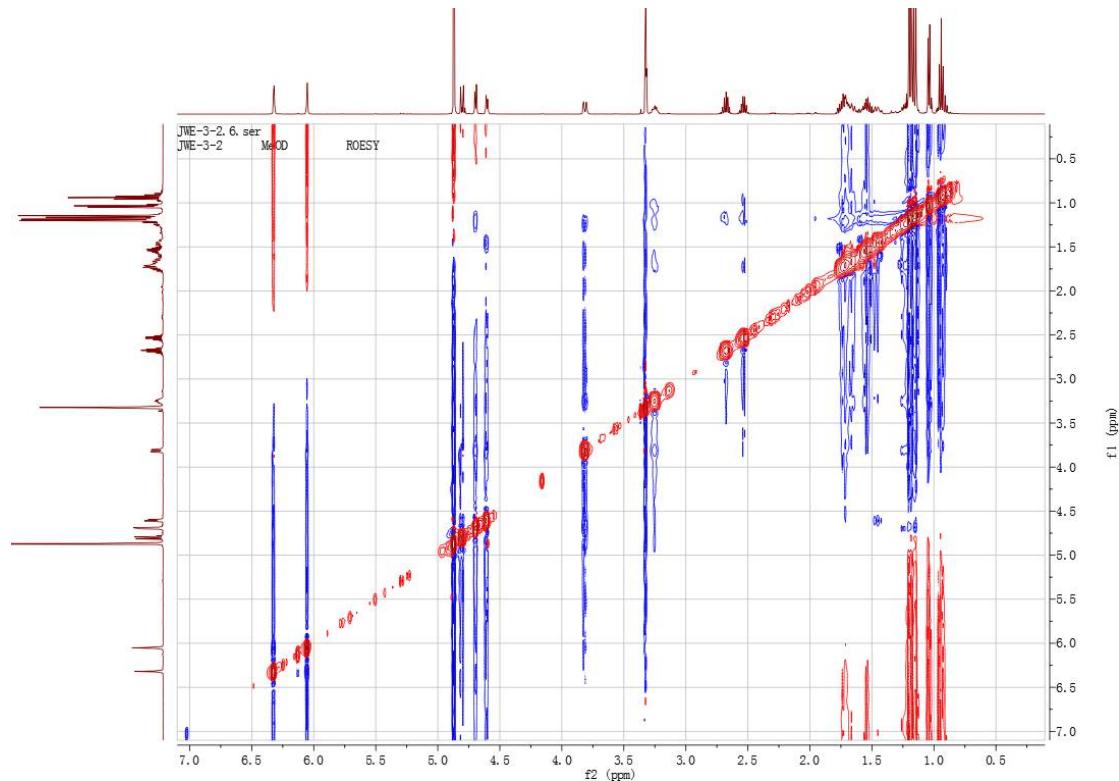
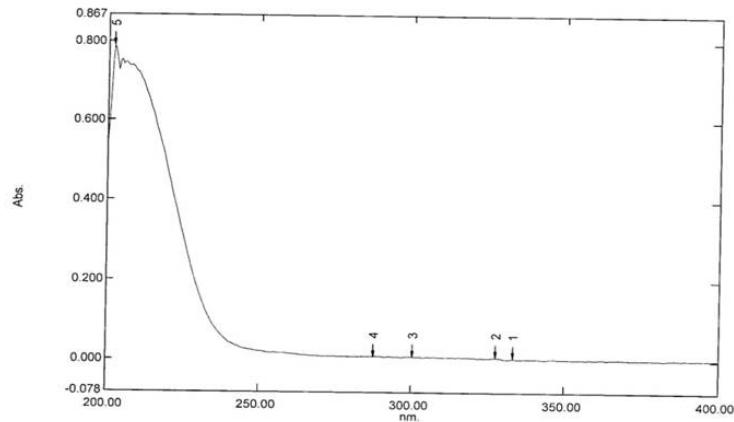


Fig. S7.6 ROESY spectrum (500 MHz) of incaspitolide B₂ (7) in CD₃OD

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Data Set: Storage 143656 - RawData - D:\2015\JWE-3-2.spc



No.	P/V	Wavelength	Abs.	Description
1	(P)	333.50	0.004	
2	(P)	327.50	0.006	
3	(P)	300.50	0.008	
4	(P)	287.50	0.009	
5	(P)	202.00	0.788	

Fig. S7.7 UV spectrum of incaspitolide B₂ (7)

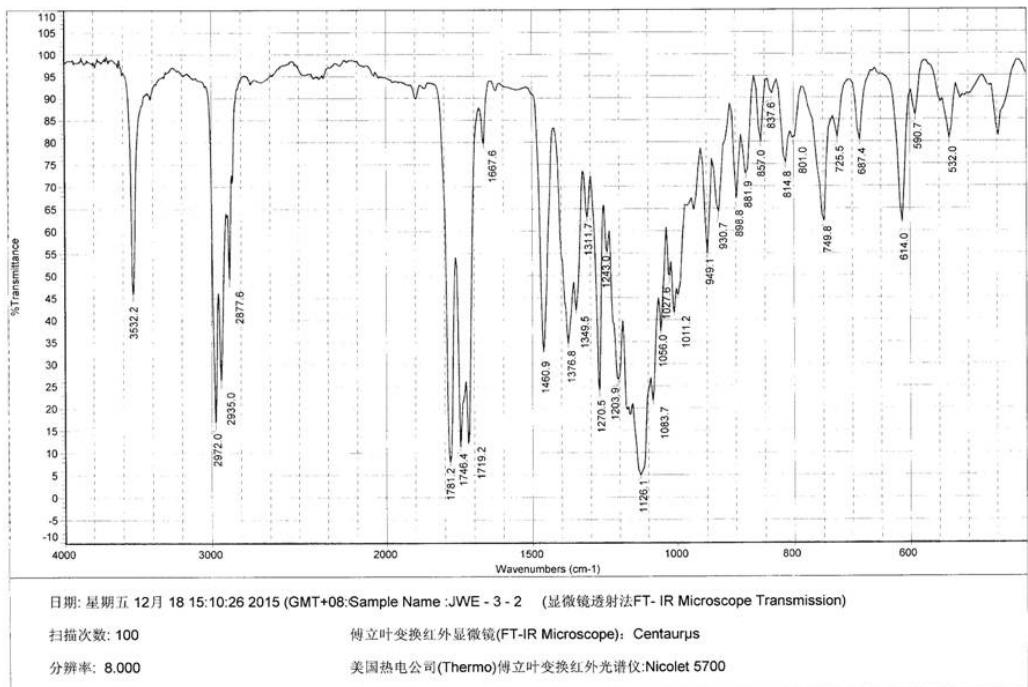


Fig. S7.8 IR spectrum of incaspitolide B₂ (7)

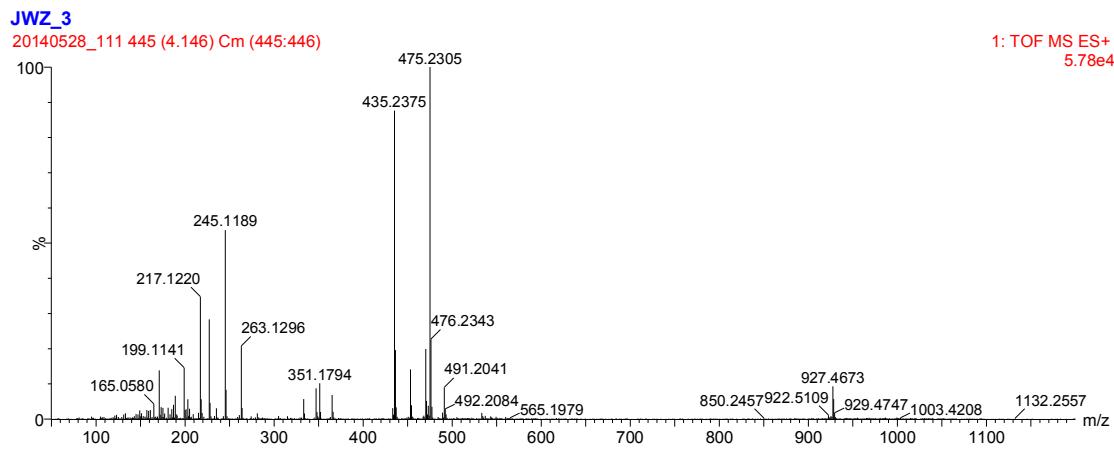


Fig. S7.9 HRESIMS spectrum of incaspitolide B₂ (7)

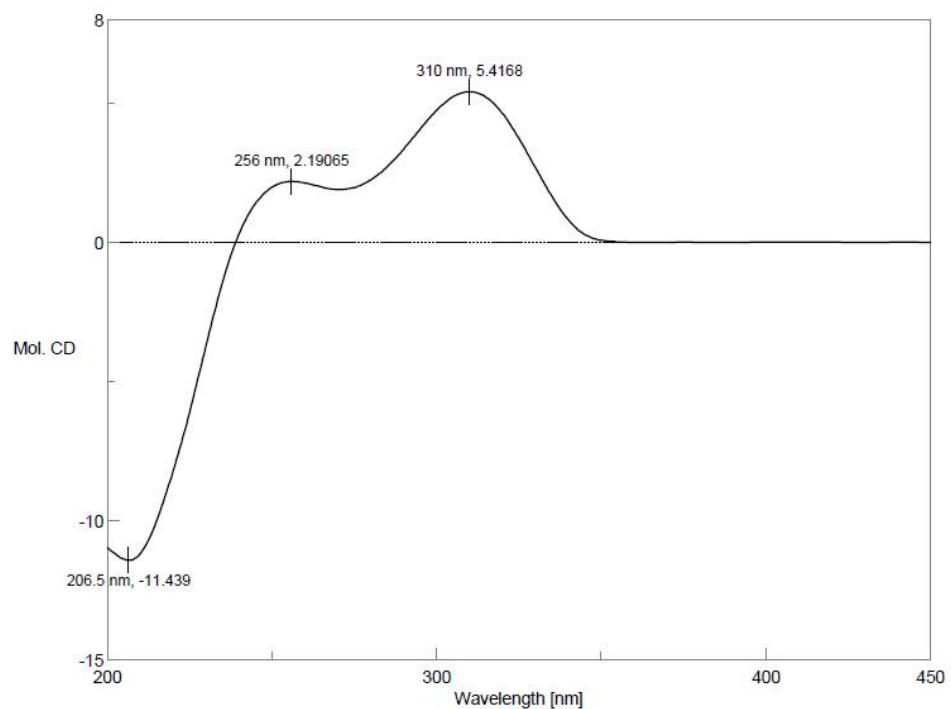


Fig. S7.10 CD spectrum of incaspitolide B₂ (7)

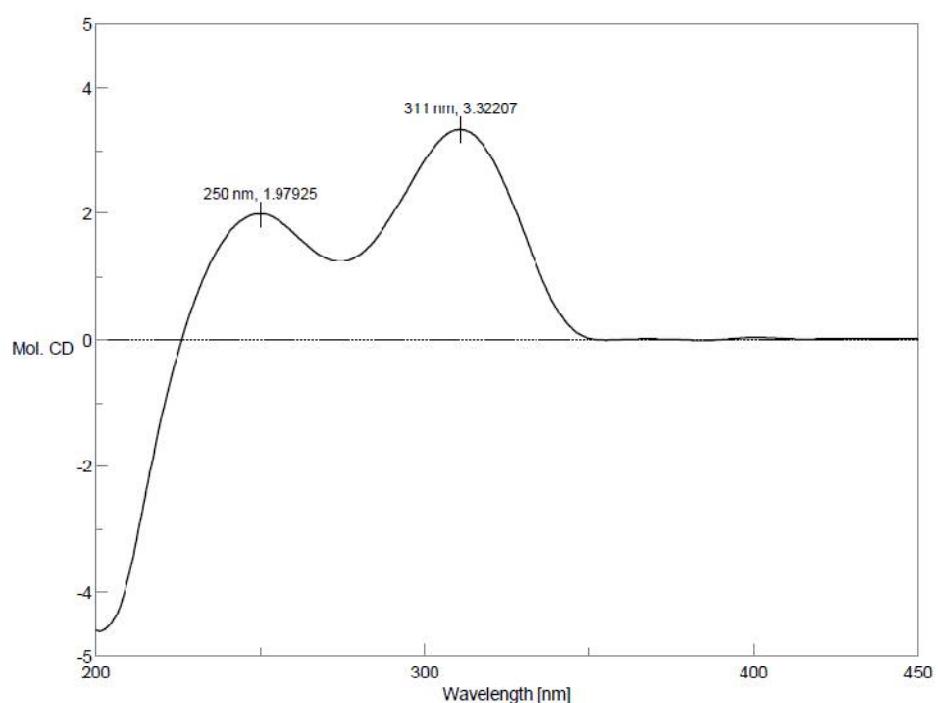


Fig. S8.1 CD spectrum of compound 8

Table S8.2 ^1H and ^{13}C NMR data of compound **8** (δ in CD_3OD ; J in Hz).

position	8	
	δ_{H}	δ_{C}
1	1.68 o, 1.23 m	22.9
2	1.56 m, 1.45 m	37.7
3	1.68 o, 1.68 o	34.5
4		73.8
5	4.68 d (6.0)	78.6
6	4.60 dd (6.0, 1.5)	73.3
7	3.85 dd (11.5, 1.5)	46.3
8	4.92 d (11.5)	79.8
9		213.5
10	3.27 m	42.7
11		134.9
12		170.7
13	6.26 d (1.5), 5.96 d (1.5)	127.4
14	1.00 d (7.0)	20.9
15	1.14 s	24.8
1'		178.7
2'	2.66 m	34.9
3'	1.17 d (7.0)	19.2
4'	1.15 d (7.0)	19.2
1''		167.1
2''		127.4
3''	6.31 q (6.5)	143.1
4''	2.00 br s	20.7
5''	1.99 dq (6.5, 1.5)	16.1

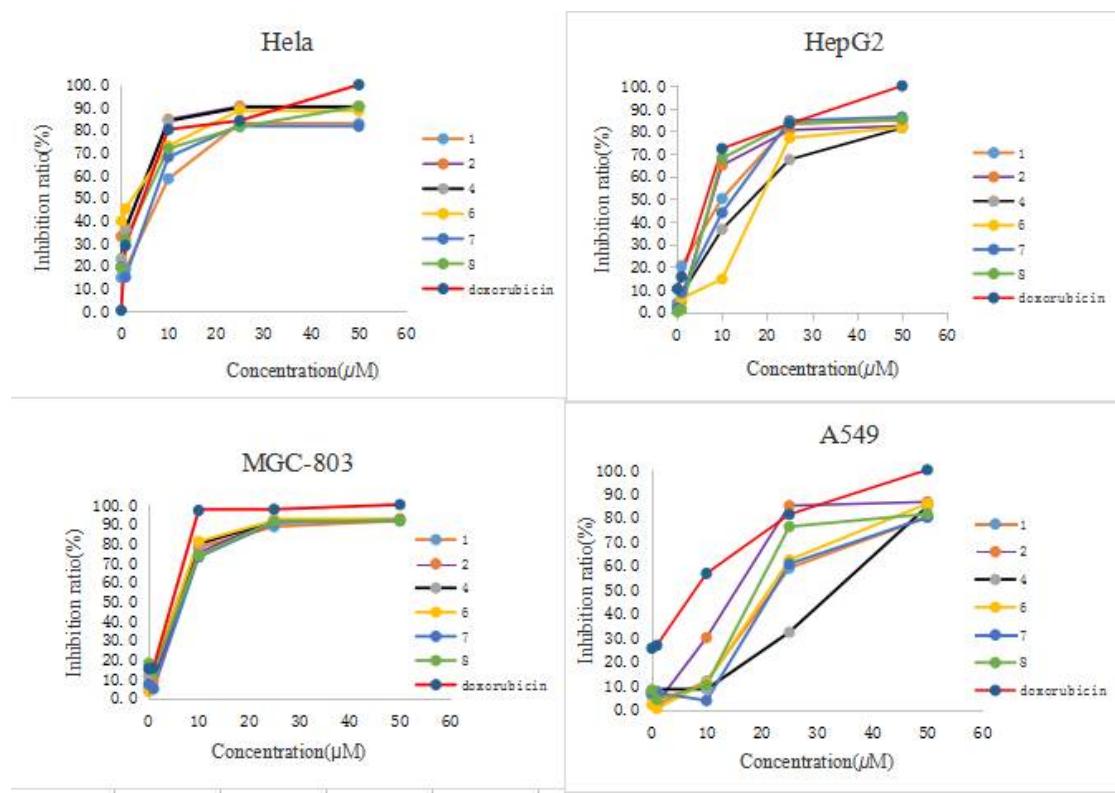


Figure S9 The concentration-response curves of compounds **1**, **2**, **4**, **6–8**, and doxorubicin (positive control) for cytotoxicity against four human tumor cell lines.